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INERTIAL CASCADE IMPACTOR SUBSTRATE MEDIA FOR FLUE GAS SAMPLING

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16. ABSTRACT

The report summarizes Southern Research Institute's experience with greases and glass fiber filter material used as collection substrates in inertial cascade impactors. Available greases and glass fiber filter media have been tested to determine which are most suitable for flue gas sampling. Greases are probably not useful at above 177 C. For higher temperatures, glass fiber filter material can be used. Of 19 greases tested by heating in the laboratory and by exposure to flue gas in the field, only Apiezon H grease performed satisfactorily at above 149 C. In experiments to evaluate the use of filter materials as impactor substrates, mass increased as a result of exposure to flue gas for all of the fiber media tested. Laboratory and field studies are described which were directed toward developing a method to passivate glass fiber filter material to SOx induced mass gains. These studies indicate that a H2SO4 wash, followed by a thorough distilled water/isopropanol rinse, drying, and baking, augmented by in situ conditioning, offers the best hope for reducing SOx induced mass gains. Reeve Angel 934AH glass fiber filter material performed best among the media tested.

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ABSTRACT

This report summarizes Southern Research Institute's experience with greases and glass fiber filter material used as collection substrates in inertial cascade impactors.

Tests have been performed to ascertain which of the available greases and glass fiber filter media are most suitable for flue gas sampling. Greases are probably not useful for temperatures above 177°C (350°F). For higher temperatures glass fiber filter material can be used.

Of nineteen greases tested, by heating in the laboratory and by exposure to flue gas in the field, only Apiezon H grease was found to perform satisfactorily at temperatures above 149°C (300°F).

In experiments designed to evaluate the use of filter materials as impactor substrates it was found that mass increases occurred as a result of exposure to flue gas for all of the fiber media tested. Laboratory and field studies are described which were directed toward development of a method by which glass fiber filter material can be passivated to SO_x induced mass gains. These studies indicate that a H_2SO_4 wash followed by a thorough distilled water and isopropanol rinse, drying, and baking, augmented by in situ conditioning, offers the best hope for reduction of SO_x induced mass gains. Reeve Angel 934AH glass fiber filter material performed best among the media tested.

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SECTION 1

INTRODUCTION

Cascade impactors are widely used to determine particle size distributions in air pollution control device research programs. In these research programs a large variety of flue streams are encountered with temperatures ranging from ambient to around 370°C (700°F). Gas analyses show that many of these sources contain some SO_x components, particularly those associated with fossil fuel fired boilers.

Most impactors have collection stages which are too heavy to obtain accurate measurements of the mass of the particles collected in each size fraction. Weighing accuracy can be improved by covering the stage with lightweight collection substrate made of aluminum foil, teflon, glass fiber filter material, or other suitable lightweight materials, depending upon the particular application. Some manufacturers now furnish lightweight inserts to be placed over the collection stages. With such arrangements it is possible to collect enough material on each stage to make an accurate determination of the mass collected and avoid overloading the stage. If the stage is overloaded, once deposited particulate matter can be reentrained and deposited on another stage or the back-up filter and lead to erroneous results.

Substrate materials may also serve the purpose of changing the surface characteristics from those of a bare metal or plastic to something better suited to holding particles which impact. Thus, various greases are often used, either on bare impactor plates, or, more frequently, on metal foil substrates.

Presented in this report are the results of investigations concerning the use of two classes of impactor substrates -- greased metal foils, and fiber filter materials. Tests were made under both laboratory and field conditions to evaluate each of several greases and filter materials. The general purpose of this study is to identify specific materials and handling techniques which may be used to improve the accuracy of weight measurements in impactors by reducing uncertainties arising from changes in substrate weights.

Although normal substrate preparation includes baking and desiccation before the initial weighing, it is frequently found that weight losses can occur when sampling clean air. Tests have been conducted to investigate this phenomenon in detail.[1] It was found that with careful handling, weight loss per glass fiber substrate for Andersen impactors can be kept below 0.1 mg. This loss is attributed to loss of fibers which stick to seals within the impactor and to "superdrying" when sampling hot, dry air. Weight losses of 0.1 mg are small compared to most stage catches when sampling particulate matter, and thus are within an acceptable range for sampling errors.

A more significant problem is excessive weight gain of the glass fiber material itself due to gas phase reactions. These reactions appear to be caused by the SO_x component in flue gases. A series of studies was thus directed toward developing procedures to passivate glass fiber materials against the effect of SO_x components in flue gases.

Although greases offer good impedance to particle bounce on substrates, they are subject to temperature limitations. Sampling clean, hot air while using greased substrates may result in severe weight losses. These losses appear to result from one or more of several mechanisms which may include continued loss of volatile components, erosion of grease by the action of the gas jet in the impactor, and occassional flow of grease from the substrate to other surfaces within the impactor. In addition, chemical reactions may play a role in some cases. Occasionally, some of the weight lost on upper impactor stages has been found to reappear on a backup filter, which is an indication that the grease has been blown off the collection surface or has chemically reacted to form a "smoke" which was then collected by the backup filter.

In this report on evaluation of greases and an evaluation of filter media are presented separately. A further division within each of these sections is made between preliminary laboratory screening tests and field studies. Under the latter headings, detailed notes and information are presented for each substrate medium.

SECTION 2

EXPERIMENTAL PROCEDURES

In order to evaluate the suitability of various greases and filter materials for application in cascade impactors, an investigation, based on a program of preliminary laboratory screening tests, followed up by field testing, was carried out. The laboratory tests were designed to characterize the behavior of the materials being studied at elevated temperatures, and to identify problems associated with specific materials. In the field studies, direct exposure to flue gas was used as a means of examining the behavior of greases and filter substrate mats in situ.

EVALUATION OF GREASES

The use of greases, usually on metal foil substrates, is an important method for controlling particle bounce and scouring effects on impaction surfaces. The principal difficulty with the use of greases in general is the effect of elevated temperature on their physical and chemical characteristics. Greases are complex organic systems which may break down at high temperatures. A decrease in viscosity might occur, in which case a grease could flow easily from the surface on which it was applied.

Preliminary Laboratory Screening Tests

Since the principal concern regarding the use of greases on impactor substrates is stability at elevated temperatures, the laboratory screening tests consisted of heating samples in an oven for periods of several hours, followed by examination of the materials for changes in consistency, color and mass.

Under EPA Contract 68-02-0273 laboratory tests were made on the nineteen greases listed in Table I. Quantities ranging from about 60 to 300 mg of undiluted grease were placed on a metal foil and heated to 160°C (320°F). Tables II and III show the experimental results. Six greases were determined to be inert enough at elevated temperature (160°C, 320°F) to warrant field tests with filtered flue gas. These were Dow Corning Molykote 111, Dow Corning 11 Compound, Dow Corning High Vacuum Grease, Apiezon L and Dow Corning 200 Fluid.

Under the current contract, Apiezon L grease was further tested in the laboratory by baking prepared MRI impactor substrate sets for extended times at 195°C (383°F). These

Table I

Greases Tested for Mass Change

1. Vaseline - Cheseborough-Pond's Inc., Greenwich, Conn. 06830
2. Molykote 111 Compound - Dow Corning Corp., Midland, Michigan 48640
3. Stopcock Grease - Dow Corning Corp., Midland, Michigan 48640
4. Dow Corning 11 Compound - Dow Corning Corp., Midland, Michigan 48640
5. Polyethylene Glycol 600 (Jefferson) - Applied Science Laboratories, Inc., State College, Pa. 16801
6. High Vacuum Grease - Dow Corning Corp., Midland, Michigan 48640
7. UCW:98 - Applied Sciences Laboratories, Inc., State College, Pa. 16801
8. Apeizon L - Applied Sciences Laboratories, Inc., State College, Pa. 16801
9. Kiln Bearing Grease - Citadel Cement Co., Birmingham, Ala.
10. 200 Fluid - Dow Corning Corp., Midland, Mich. 48640
11. Fluorolube GR-362 - Hooker Chemicals and Plastics Corp., Niagara Falls, N.Y.
12. Fluorolube GR-544 - Hooker Chemicals and Plastics Corp., Niagara Falls, N.Y.
13. Fluorolube GR-470 - Hooker Chemicals and Plastics Corp., Niagara Falls, N.Y.
14. Fluorolube GR-290 - Hooker Chemicals and Plastics Corp., Niagara Falls, N.Y.
15. Fluorolube GR-660 - Hooker Chemicals and Plastics Corp., Niagara Falls, N.Y.
16. Carbowax 1000 - Applied Sciences Laboratories, Inc., State College, Pa. 16801
17. Carbowax 4000 - Applied Sciences Laboratories, Inc., State College, Pa. 16801
18. Carbowax 20M - Applied Sciences Laboratories, Inc., State College, Pa. 16801
19. STP Oil Treatment - STP Corporation, Fort Lauderdale, Fla. 33310

Table II

Cumulative Mass Change
4 Consecutive Hourly Bakings at 160°C

Grease	Original Shape	Original Consistency	Original Color	Initial Mass (mg)	1 hr 160°C		2 hr 160°C		3 hr 160°C		4 hr 160°C		Final Shape, Consistency, Color
					Mass Change	% Change	Mass Change	% Change	Mass Change	% Change	Mass Change	% Change	
1.	Pointed	Malleable	Frosty White	70.18	1.14	1.62	2.73	3.89	4.57	6.51	5.50	7.84	Flat, Dry, Tacky, Yellow
2.	Pointed	Malleable	Frosty White	92.75	0.23	0.25	0.30	0.32	0.34	0.37	0.36	0.39	Pointed, Malleable, Frosty
3.	Pointed	Malleable	Frosty White	89.97	0.45	0.50	0.53	0.59	0.57	0.63	0.57	0.63	Rounded, Malleable, Blue
4.	Pointed	Malleable	Frosty White	75.99	0.25	0.33	0.31	0.41	0.37	0.49	0.39	0.51	Rounded, Malleable, Frosty
5.	Flat	Liquid	Clear	86.34	7.79	9.02	13.58	15.73	22.78	26.38	28.69	33.23	Flat, Dry, Clear
6.	Pointed	Malleable	Frosty White	125.24	0.66	0.53	0.74	0.59	0.81	0.65	0.81	0.65	Rounded, Malleable, Frosty
7.	Rounded	Highly Viscous	Clear	62.98	0.53	0.84	0.68	1.08	0.81	1.29	0.82	1.30	Flat, Tacky, Clear
8.	Pointed	Malleable	Opaque Orange	79.50	+0.09	0.11	+0.06	0.08	+0.08	0.10	+0.12	0.15	Flat, Tacky, Yellow
9.	Pointed	Malleable	Opaque Orange	120.52	9.96	8.26	12.95	10.75	16.97	14.08	18.72	15.53	Flat, Dry, Tacky, Brown
10.	Flat	Highly Viscous	Clear	173.55	20.72	11.94	20.92	12.05	20.96	12.08	21.09	12.15	Flat, Malleable, Frosty
11.	Rounded	Malleable	Clear	229.25	102.76	44.82	137.59	60.01	159.28	69.48	167.90	73.24	Pointed, Malleable, Clear
12.	Rounded	Malleable	Clear	300.04	31.54	10.51	51.97	17.32	71.50	23.83	82.67	27.55	Rounded, Malleable, Clear
13.	Pointed	Malleable	Frosty White	139.48	53.26	38.19	70.07	50.24	80.12	57.44	84.61	60.66	Round, Dry, White, Opaque
14.	Pointed	Malleable	Frosty White	161.42	24.81	15.39	41.56	25.75	56.17	34.80	64.36	39.87	Flat, Dry, Hard
15.	Pointed	Malleable	Frosty White	137.01	11.39	8.31	20.33	14.84	28.70	20.95	33.20	24.22	Flat, Dry, Clear
16.	Pointed	Slightly Viscous	Frosty White	131.84	11.49	8.72	25.03	18.99	33.13	25.13	39.37	29.86	Flat, Dry, Clear
17.	Pointed	Solid Crystals	Opaque White	54.30	7.08	13.04	14.49	26.69	18.07	33.28	20.89	38.47	Flat, Dry, Clear
18.	Pointed	Solid Crystals	Opaque Brown	87.15	2.10	2.41	8.15	9.35	13.63	15.64	18.48	21.20	Flat, Tacky, Clear
19.	Flat	Viscous	Clear Brown	69.12	3.21	4.64	5.41	7.83	7.48	10.82	9.33	13.50	Flat, Viscous, Clear Brown
Metal Controls													
Standard Aluminum				73.81	0.08		0.06		0.08		0.08		
Standard Ferrule				69.56	0.02		+0.01		0.01		0.01		

Table III

Mass Change After Continuous Baking at 160°C

Grease	Initial Mass (mg)	4 hr 160°C Mass Change	4 hr 160°C % Change
1.	69.43	- 5.91	8.51
*2.	97.09	- 0.09	0.09
*3.	49.82	- 0.20	0.40
*4.	41.07	- 0.25	0.61
5.	66.75	- 64.85	97.15
*6.	49.55	+ 0.01	0.02
7.	57.37	- 0.37	0.64
*8.	34.77	0.00	0.00
9.	50.65	- 11.82	23.33
*10.	63.25	- 0.47	0.74
11.	104.30	- 84.41	80.93
12.	101.32	- 41.71	41.17
13.	161.31	-100.53	62.31
14.	133.89	- 57.01	42.58
15.	120.59	- 36.90	30.60
16.	81.01	- 49.62	61.25
17.	60.16	- 44.54	74.04
18.	49.60	- 24.65	49.70
19.	81.41	- 17.74	21.79

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*These qualified for further field testing.

substrates were prepared by dropping or painting on 10% Apiezon L-Toluene solutions. Table IV shows the results of this study. The conclusion of this study was that at elevated temperatures (195°C, 383°F) Apiezon L grease was not a good candidate for substrate coating because the thinly coated foils developed hard enamel-like surfaces. Near the edges of more heavily coated substrates grease accumulated, and in these deposits no such hard coating was observed. However, these deposits had changed color.

Field Studies

Dow Corning Greases

Under Contract 68-02-0273 (EPA) limited tests were performed on Dow Corning Molykote 111 and Dow Corning Silicone High Vacuum Grease used as impactor substrate coatings. Substrates of a University of Washington Source Test Cascade Impactor were coated with these greases and used to sample flue gas for one hour at a steam plant. The flue gas was first filtered using a Gelman 47 mm Type A glass fiber filter. Table V shows the results of this study. While the Dow Corning Molykote 111 was much more stable than the silicone high vacuum grease a significant weight loss did occur on each stage. Both back-up filters had an abnormally high weight gain which was probably due to bake-off or scouring of material from the greases. As a result of this test it was decided that the Dow Corning greases are not suitable for flue gas sampling. Note that only two out of the five Dow Corning greases were tested. Therefore our conclusion represents a generalization. However, the physical properties of these greases are similar enough to make it reasonable.

Apiezon Greases

Under EPA Contract 68-02-2131 the behavior of Apiezon H, L, M, N, and T greases in filtered flue gas streams were tested at another steam plant. For the flue gas exposure tests, substrates were made using either aluminum or stainless steel foils. The greases were applied by dropping or painting on a 5% grease-toluene solution of Apiezon H, M, N, or T or a 10% grease-toluene solution of Apiezon L. After application the substrates were dried in an oven at 165°C (329°F) for several hours.

Two University of Washington Source Test Cascade Impactors were used in these tests. Gelman 47 mm prefilters were used to precede the impactors so that the substrates would be subjected only to the flue gas. All impactor runs were made horizontally. The impactor was allowed to warm-up for 30 to 45 minutes in the flue gas stream before each run began. The runs were 2 to 2.5 hours in duration. The impactor flowrate was 0.7 ACFM, monitored with an orifice and flowmeter. Table VI summarizes the results of these runs. Note that each substrate set was subjected to the 149°C (300°F) flue gas for approximately 2.5 hours.

TABLE IV

Laboratory Bakeout of Apiezon L. Greased Substrates

Preparation	Mass (mg) of Grease on Plate During Bakeout at 195°C (383°F)						
	2.5 hours	7 hours	Δ(mg)	Δ(%)	22 hours	Δ(mg)	Δ(%)
1. Paint on solution to visibly wet surface (foil)	11.66	11.31	-0.35	-3.0	10.75	-0.91	-7.8
2. 20 drops of solution (foil)	47.92	46.95	-0.97	-2.0	45.70	-2.22	-4.6
3. 15 drops of solution (MRI plate)	23.30	22.10	-1.20	-5.2	20.70	-2.60	-11.2
4. Paint on film one time (MRI plate)	2.50	2.20	-0.30	-12.0	2.00	-0.50	-20.0
5. Paint on film 3 times (MRI plate)	6.80	6.10	-0.70	-10.3	5.20	-1.60	-23.5
6. No treatment (MRI plate)	0.5*	0.3	-0.2	-40.0	0.1	-0.4	-80.0

Appearance of grease after 2.5 hours: Yellow-orange film of grease on heavily coated surfaces

Appearance of grease after 22 hours: Orange-gold-brown color. Tacky sticky, still "greasy" on heavily coated parts (near edge). Thinly coated parts have hard enamel-like surfaces.

*Mass change of clean MRI plate after 2.5 hours bakeout in oven with other greases.

TABLE V

Mass Changes of Dow Corning Silicone Greases

SUBSTRATE	DOW MOLYKOTE 111 COMPOUND	DOW SILICONE HIGH VACUUM GREASE
TEMP	149°C (300°F)	138°C (280°F)
FLOWRATE	0.46 ACFM	0.45 ACFM
SAMPLE DURATION	60 MIN.	60 MIN.
STAGE	MASS CHANGE (mg)	
1	-1.1	-4.06
2	-0.74	-1.74
3	-0.34	-3.60
4	-0.36	-3.76
5	-0.46	-1.32
6	-0.32	-1.90
7	-0.46	-0.64
Filter	+1.86	+2.68
AVERAGE LOSS PER STAGE	0.54 mg	2.43 mg
NET TOTAL LOSS	1.92	14.34 mg

FILTERS - UNPRECONDITIONED GELMAN TYPE A (OLD TYPE)
EXPECTED FILTER MASS CHANGE ≃ 0.2 mg

TABLE VI

Summary of Flue Gas Test Results. The Flue Gas Temperature Averages About 149°C (300°F) for All Tests. Impactor Flow Rate Was 0.7 ACFM.

Substrate Set	1 (Apiezon L)			2 (Apiezon L)			3 (Apiezon M)			4 (Apiezon T)			5 (Apiezon H)[5]			6 (Apiezon N)		
	Grease on Substrate (mg)	Mass (mg)	Change (%)	Grease on Substrate (mg)	Mass (mg)	Change (%)	Grease on Substrate (mg)	Mass (mg)	Change (%)	Grease on Substrate (mg)	Mass (mg)	Change (%)	Grease on Substrate (mg)	Mass (mg)	Change (%)	Grease on Substrate (mg)	Mass (mg)	Change (%)
Stage																		
0	Blank	+0.23	-	Blank	+0.18	-	Blank	+0.08	-	Blank	+0.04	-	Blank	+0.01	-	Blank	+0.03	-
1	25.05 D	-4.96[3]	19.8	35.28 D	-3.25[1]	9.2	21.69 D	-0.02[3]	0.1	23.68 D	-1.10[3]	4.6	41.55 D	+1.56	3.8	18.19 D	-1.52[3]	8.4
2	13.48 P	-7.29[1]	54.1	7.51 P	+0.33	4.4	3.19 P	+0.04	1.3	1.89 P	-0.25	13.2	4.18 P	+0.82	19.6	4.41 P	+0.05	1.1
3	38.33 D	-27.98[1]	73.0	37.24 D	-26.54[1]	71.3	19.03 D	-7.14[3]	37.5	3.25 P	-0.21	6.5	3.81 P	+0.80	21.0	19.00 D	-1.75	9.2
4	11.99 P	-0.64	5.3	9.14 P	+0.15	1.6	4.36 P	+0.10	2.3	24.87 D	-0.61	2.5	28.04 D	+0.44	1.6	3.37 P	-0.06	1.8
5	39.36 D	-24.18[1]	61.4	29.28 D	-13.05[1]	44.6	26.09 D	-10.11[3]	38.8	24.78 D	-1.07[3]	4.3	30.13 D	+0.53	1.8	18.79 D	-1.73[3]	9.2
6	12.06 P	-0.35	2.9	8.69 P	+0.34	3.9	7.10 P	-0.05	0.7	3.69 P	+0.09	2.4	4.87 P	-0.05	1.0	4.14 P	-0.10	2.4
Back-up	-	+0.15[2]	0.1[6]	-	+0.17[2]	0.2[6]	-	+4.40[4]	3.1[6]	-	+5.13[4]	3.6[6]	-	+4.36[4]	3.0[6]	-	+4.74	3.3[6]

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1. Grease lost to overflow, not recovered.
2. Reeve-Angel 934AH Filter Material, preconditioned.
3. Some grease lost to overflow, most recovered and deposited back on foil.
4. Gelman AE Filter Material, preconditioned. Weight gains may be partially due to flue gas reactions.
5. Substrates contaminated with ambient dust. Probably not enough to alter results significantly.
6. Percent mass change in filter calculated using final filter weight.

Note: D means that the grease-toluene mixture was dropped on with an eyedropper (20 to 25 drops).
P means that the grease-toluene mixture was painted on with a paint brush (3 to 4 times).

A problem of grease overflow occurred in some of the tests as a result of reduced grease viscosity at elevated temperature. This normally resulted in unaccounted loss of mass. Moreover, excessive loss of grease due to overflow would generally be considered sufficient grounds for rejection of the grease, since behavior leading to overflow could not be tolerated in actual use on impactor substrates.

The results of these tests are summarized below. A total of 6 substrate sets were made up: two sets for Apiezon L and one set each for the rest. Table VII gives the published physical properties of the Apiezon greases. Figure numbers refer to photographs of greased substrates.

Apiezon L (Figures 1, 2, and 3)--

Substrate sets 1 and 2 were prepared with Apiezon L grease. The substrates that were heavily coated (>20 mg of grease) developed a hard, black, enamel-like surface and the grease showed evidence of being blown away from beneath the jets. Lightly coated substrates did not develop the "enameled" appearance; however, the grease was blown away from beneath all the jets, and the grease was not hardened. Considerable grease overflow occurred during these runs; the low viscosity of Apiezon L grease at 149°C (300°F) combined with horizontal impactor operation is probably responsible for this overflow. When thick overflow deposits were found beneath the foils, these deposits remained "greasy" and were not discolored.

Apiezon M (Figures 4 and 5)--

Substrate set 3 was coated with Apiezon M grease. Apiezon M has a melting point which is nearly that of Apiezon L, although its molecular weight is much lower. (See Table VI.) This grease behaves much like Apiezon L, exhibiting vivid flow patterns beneath the jets and evidence of chemical reactivity as well as overflow onto the substrate support plate. After exposure to the flue gas stream, this grease solidified at room temperature into a black, waxy solid. Figure 5 shows where all the grease had been scoured from directly beneath the jets on stage 4.

Apiezon T (Figures 6 and 7)--

Substrate set 4 was prepared with Apiezon T grease. Apiezon T has a melting point of 125°C (257°F) and although this is a fairly high melting point for grease, the aluminum and stainless steel substrates showed clear jet patterns and flow of grease. The grease was slightly discolored after exposure to the flue gas but afterwards remained "greasy". Some overflow occurred.

TABLE VII

Typical Physical Properties of Some Apiezon Greases*

PROPERTY	GREASE H	GREASE L	GREASE M	GREASE N	GREASE T
Approximate melting point, °C	(a)	47	44	43	125
Specific gravity at: 20°C		0.896	0.894	0.911	0.912
30°C		0.889	0.887	0.904	0.905
Viscosity cP of molten grease at: 50°C		766	413		
100°C		62.3	29.8		
Average molecular weight		1300	950		
Coefficient of expansion per °C over 20°C-30°C		0.00076	0.00075	0.00072	0.00073
Thermal conductivity Btu in/ft² h.deg F	1.50	1.40	1.33	1.31	1.22
w/m deg C	0.216	0.202	0.192	0.189	0.176
Specific heat at 25°C: cal/g	0.42	(b)	(b)	(b)	(b)
Joule/g	1.7	(b)	(b)	(b)	(b)
Latent heat of fusion, cal/g		15.1	18.7	15.0	
fusion peak, °C		32	34	31	
Volume resistivity, ohm-cm		1.2×10^{16}	2.6×10^{16}	2.0×10^{16}	3.3×10^{12}
Permittivity		2.3	2.1	2.3	2.3
Loss tangent		less than 0.0001			
Surface breakdown, kV at flash-over		24	28	27	24
Electric strength, volts/mil		730	850	820	730

a. Grease H does not melt at high temperatures and consequently many of the above physical properties cannot readily be measured.

b. Specific heats of Greases L, M, N and T cannot be measured as their fusion peaks are too close to room temperature.

*From Bulletin 43a, Apiezon Oils, Grease and Waxes, James G. Biddle Co., Plymouth Meeting, PA 19462.

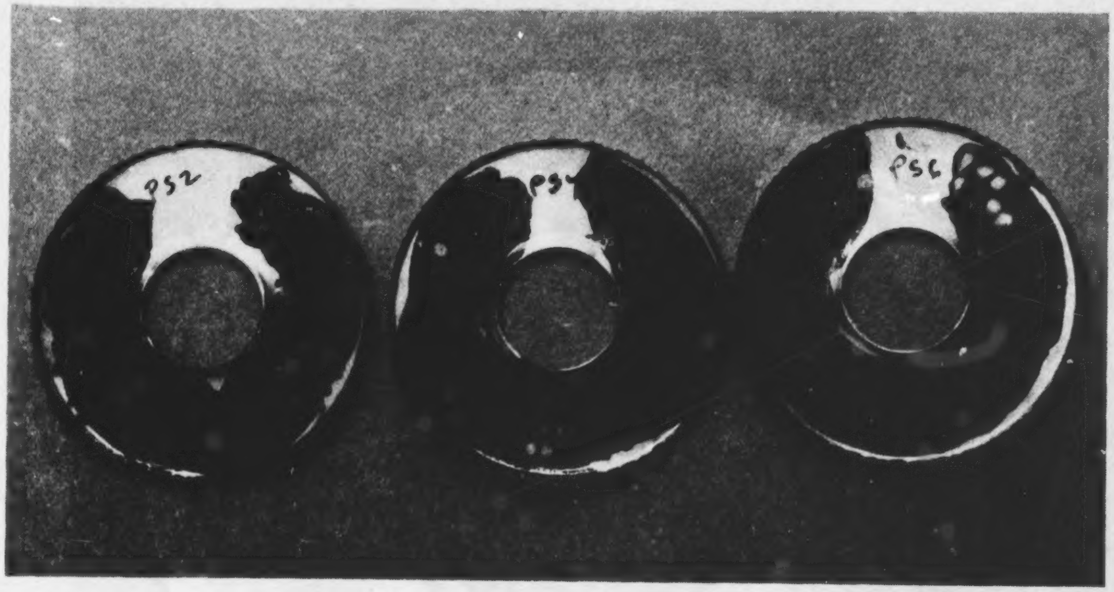

Figure 1. Apiezon L greased substrates, grease-toluene mixture dropped on. Stages 2, 4, and 6. Stainless steel substrates.

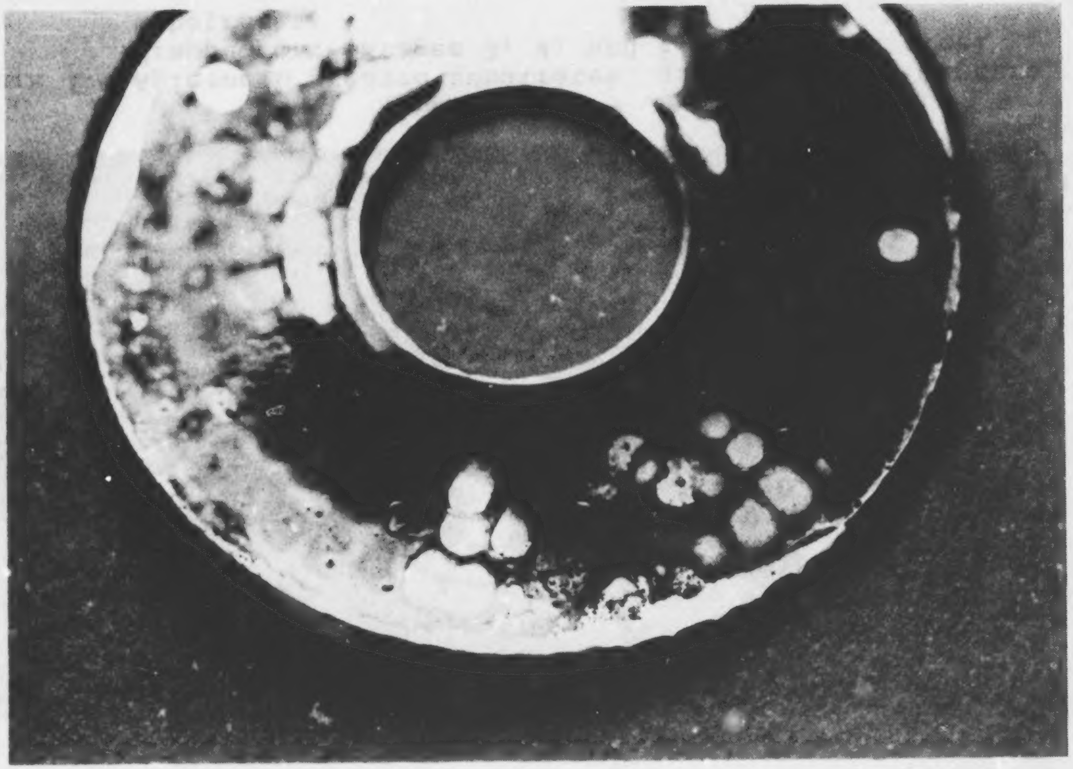

Figure 2. Apiezon L greased substrate, grease-toluene mixture dropped on. Closeup of Stage 4. Stainless steel substrate.

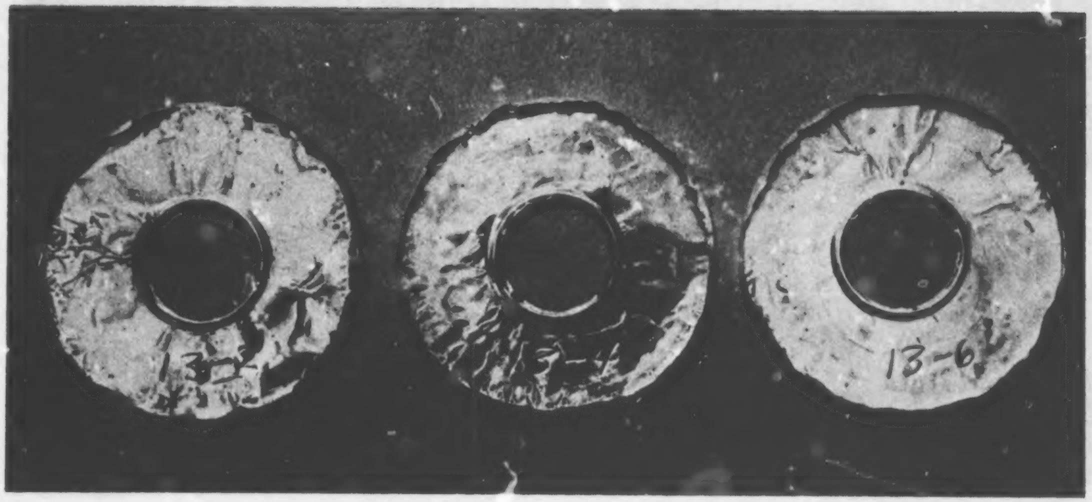

Figure 3. Apiezon L greased substrates, grease-toluene mixture painted on. Aluminum foil substrates.

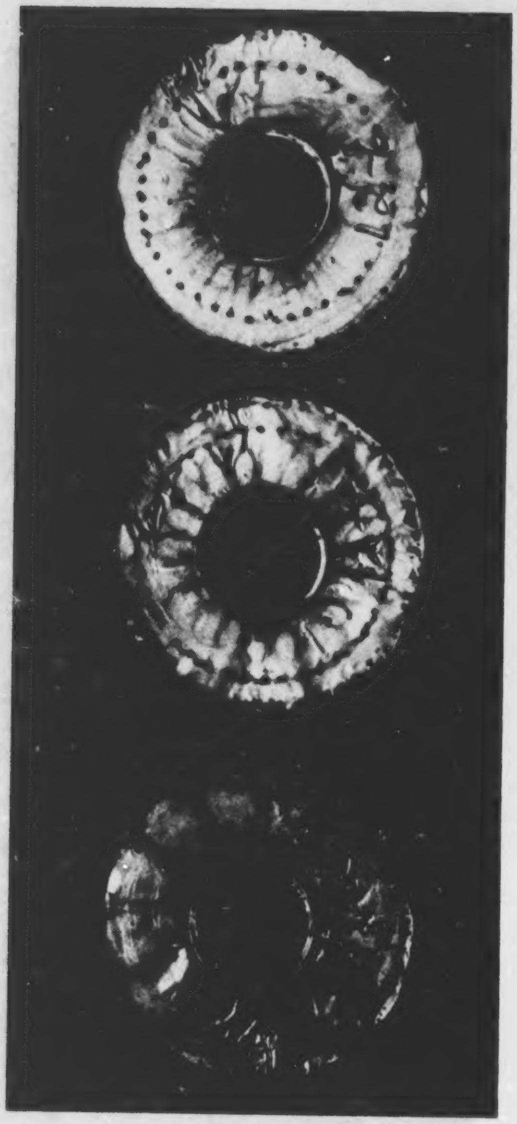

Figure 4. Apiezon M greased substrates, grease-toluene mixture painted on. Aluminum foil substrates.

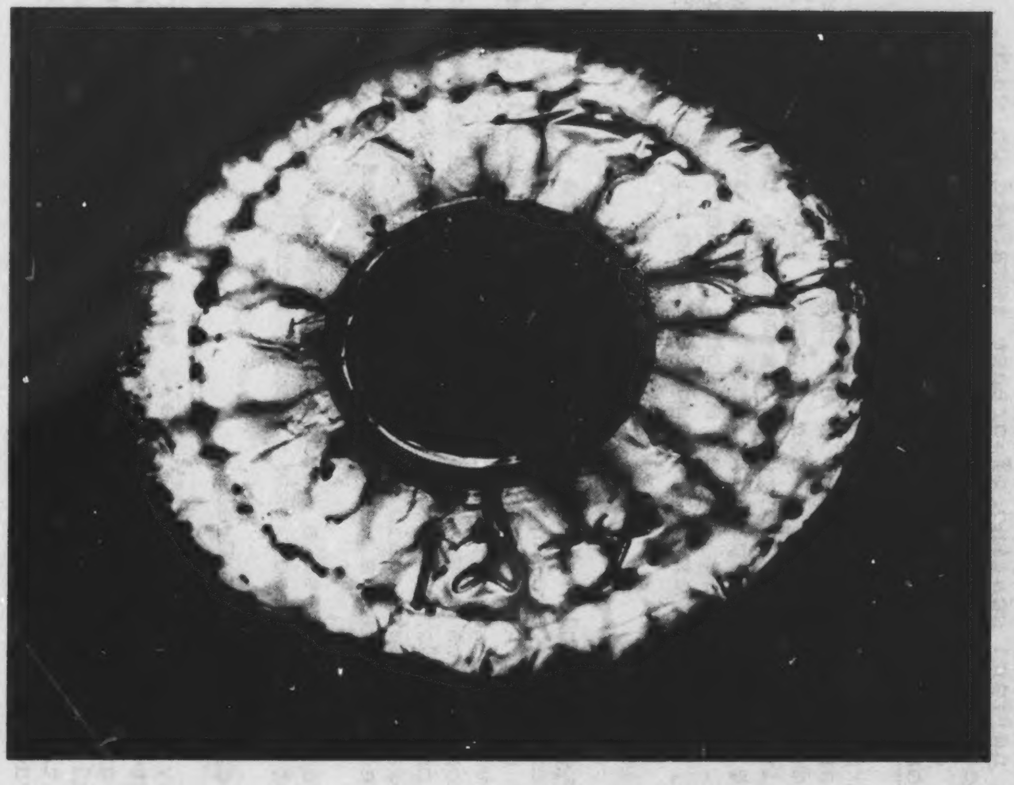

Figure 5. Apiezon M greased substrate, grease-toluene mixture painted on. Closeup of Stage 4. Aluminum foil substrate.

Apiezon H (Figure 8)--

Substrate set 5 was made up with Apiezon H grease. This grease melts above 250°C (482°F) and during this test showed no signs of having melted. Some discoloration took place but the grease remained "greasy". The discoloration could be partially due to contamination. A thin layer of room dust (∿20 μm-50 μm diameter) appears uniformly distributed over all these substrates. This could have occurred as the substrates were being prepared, as they were being unloaded or during weighing. Thus the nearly uniform weight gain of these substrates may be connected with this contamination. Jet patterns were barely visible on some stages; however, a uniform coating of grease remained on each. No overflow of the grease was observed.

Apiezon N (Figure 9)--

Substrate set 6 was prepared with Apiezon N grease. Apiezon N is similar to Apiezon L and M in that it melts between 40°C (104°F) and 50°C (122°F). When exposed to 149°C (300°F) flue gas streams this grease is scoured from beneath the jets so that none would be left to catch particulate. Grease overflow occurred on two stages. The grease showed slight evidence of having reacted chemically, turning very dark and becoming slightly waxy in places.

Back-Up Filters

Two types of glass fiber back-up filters were used in the flue gas tests. Reeve Angel 934AH was used with the Apiezon L grease and Gelman AE was used with the rest. Both types of filters took on a visibly gray color after each test. This may be due to volatile components being driven off the substrates. However, this same coloration was noticed with the Apiezon H grease which had an overall weight gain. We have seen this coloration before when greased substrates were used in field tests.

On the basis of this study we concluded that Apiezon H grease is the best available of the greases tested.

Field Use of Apiezon H Grease

In May of 1976 during a field test at a steam plant, University of Washington (UW) Source Test Cascade Impactors were used with Apiezon H greased substrates. Flue temperatures averaged 124°F (255°F) and run times were 30 minutes. All runs were made at the outlet of the electrostatic precipitator where SO_2 and SO_3 concentrations averaged 450 ppm and 0.8 ppm respectively.

Blank runs were made with the UW impactors in order to determine the magnitude of weight changes that could be expected from loss or flow of the Apiezon H grease. For a blank impactor run

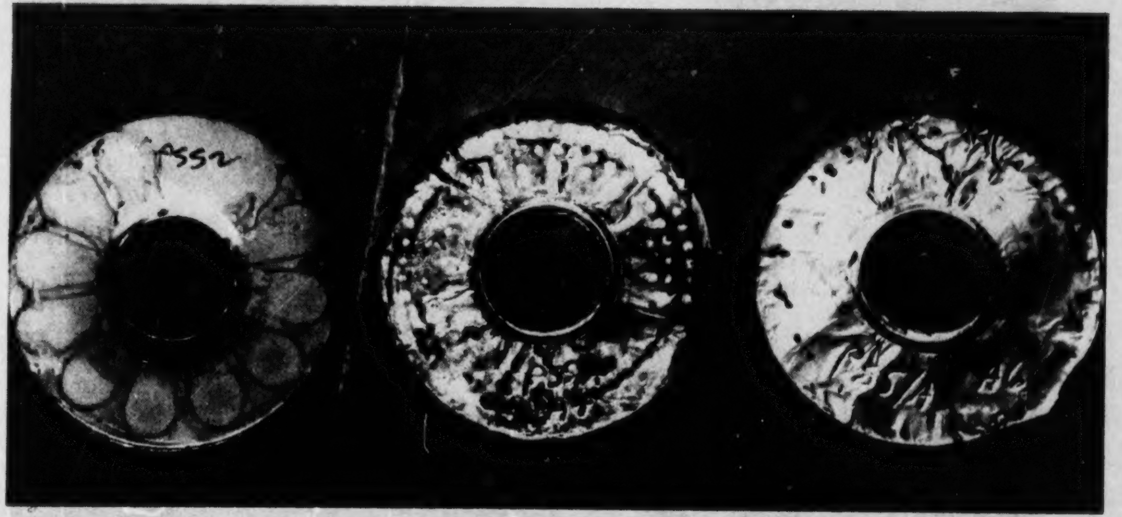

Figure 6. Apiezon T greased substrate, grease-toluene mixture painted on Stages 2 and 6, dropped on Stage 4. Stage 2 is stainless steel and Stages 4 and 6 are aluminum foil.

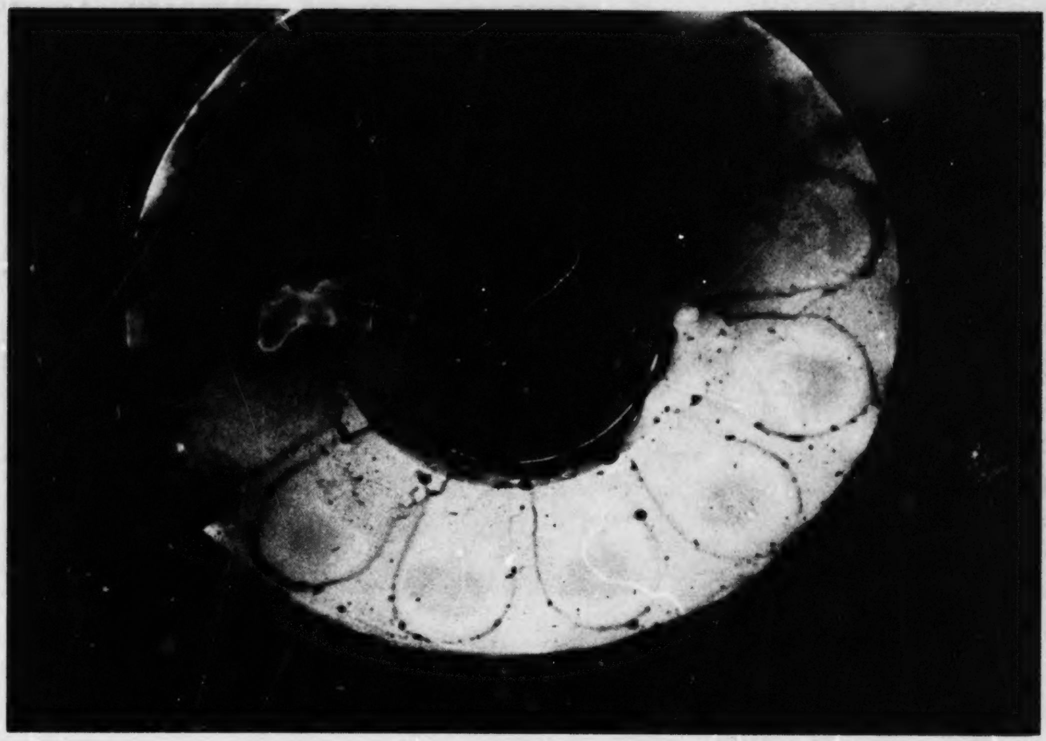

Figure 7. Apiezon T greased substrate, grease-toluene mixture painted on. Closeup of Stage 2. Stainless steel substrate.

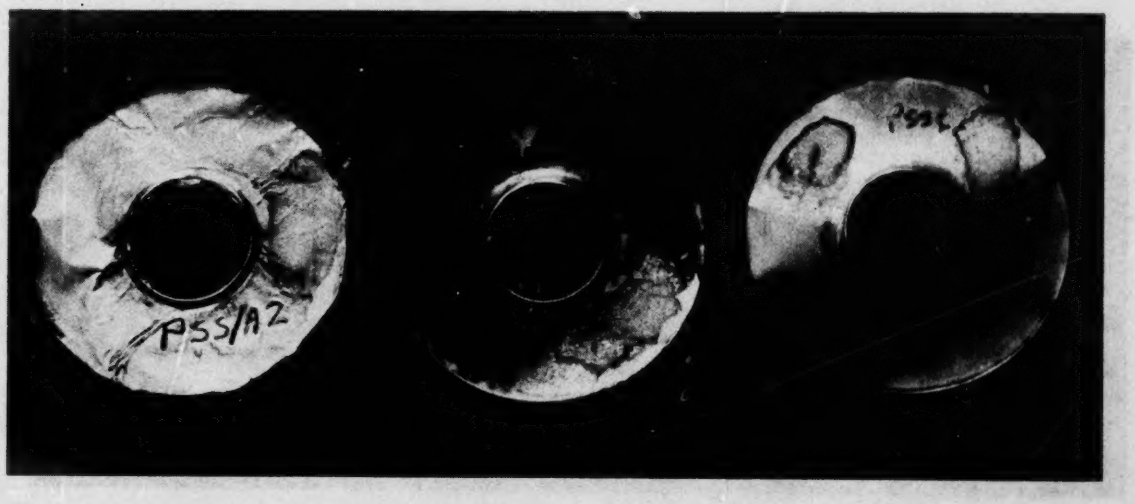

Figure 8. Apiezon H greased substrates, grease-toluene mixture painted on Stages 2 and 6, dropped on Stage 4. Stainless steel substrates.

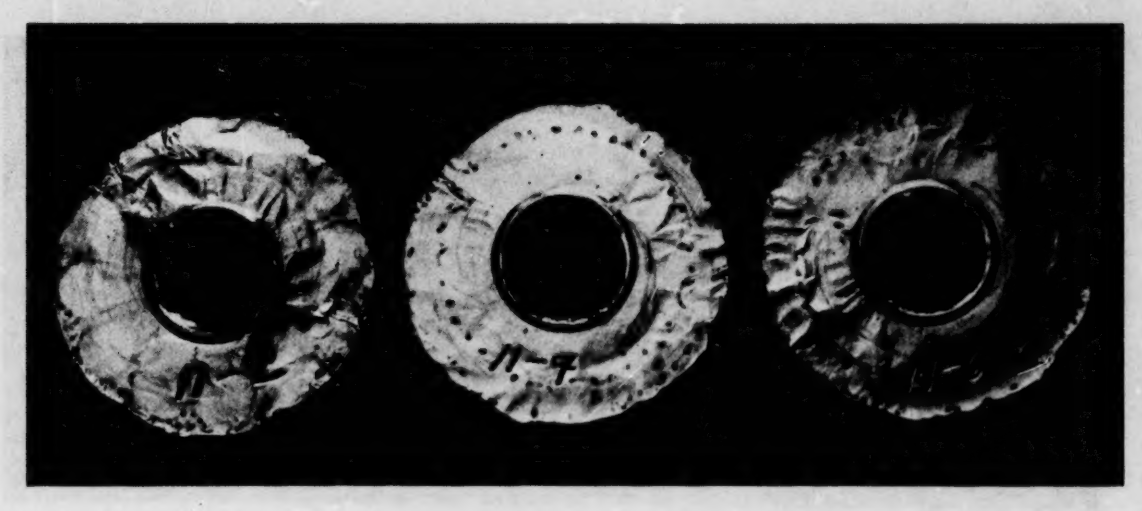

Figure 9. Apiezon N greased substrates, grease-toluene mixture painted on. Aluminum foil substrates.

the impactor is preceded by a Gelman 47 mm stainless steel filter holder fitted with a glass fiber filter. Next, the impactor is inserted into the precipitator duct and operated as in a normal run. No attempt is made to sample isokinetically, but a flow rate and run time is chosen which is typical of impactor operation.

Table VIII shows the individual stage mass changes that occurred during each blank run. The coupling between the stainless steel Gelman 47 mm filter holder and the impactor was not made of stainless steel and was subject to some corrosion. This corrosion occasionally flaked off during impactor disassembly and deposited on stage 0. Consequently, larger than average weight gains occurred on runs 4 and 5. Examination of individual substrates of the blank runs showed the following:

1. Stage 0 for each run was contaminated with some debris.
2. All substrates in run 6 were discolored.
3. The back up filters, 47 mm Reeve Angel 934AH material showed no large weight gains with the exception of run 5, and none of the filters were discolored.
4. With the exception of run 5, discoloration of the grease was infrequent and unrelated to weight gains.
5. The grease showed no sign of chemical degradation or flow for all runs.

Since these weight gains are not much different from those observed when glass fiber substrates are used (see Tables XXII and XXVI) we conclude that Apiezon H grease will be a suitable substrate material when flue temperatures do not exceed 177°C (350°F). At higher temperatures some material bakes out of the grease and weight losses occur. A limited laboratory test shows that at 288°C (550°F), Apiezon H grease loses weight and undergoes chemical change.

Summary of Results of Evaluation of Greases

Upon preliminary screening by static heating tests in the laboratory, six of the nineteen greases tested were found to have acceptable characteristics at elevated temperatures. Among those greases eliminated by these tests, large changes in mass or in consistency had occurred.

In the field tests greases were applied to metal foil impactor substrates and were subjected to a flue gas sampling procedure. Particulate matter was removed by a prefilter so that the effects of the flue gas alone on the greased substrates could be observed.

TABLE VIII

Apiezon H Blank Mass Gains for University of Washington Mark III Source Test Cascade Impactors. Coal fired power boiler source, cold side precipitator operating at 107°C (225°F), 30 minute runtime. All masses in milligrams.

Stage	Run 1 5/17/76	Run 2 5/17/76	Run 3 5/19/76	Run 4 5/20/76	Run 5 5/21/76	$\overline{x}$	σ
0	0.22	0.08	0.16	0.55	0.65	0.33	0.25
1	0.06	0.09	-0.18	0.11	0.85	0.19	0.39
2	0.04	0.01	0.04	0.10	0.64	0.17	0.27
3	-0.03	-0.11	0.09	-0.13	0.64	0.09	0.32
4	0.02	-0.08	0.09	-0.05	0.47	0.09	0.22
5	-0.06	-0.13	0.05	-0.03	0.31	0.03	0.17
6	-0.14	-0.12	0.13	-0.05	0.16	-0.004	0.14
Filter*	-0.32	-0.18	0.02	0.08	0.34	-0.01	0.25

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* Reeve Angel 934AH 47 mm disc.

As a result of the field studies it was concluded that Apiezon H grease performed best of the greases tested. Other greases studied displayed changes in consistency or a tendency to flow under the influence of the gas stream.

Further tests on Apiezon H have demonstrated that this grease is a suitable substrate material for applications where the temperature does not exceed approximately 177°C (350°F).

EVALUATION OF FILTER MEDIA FOR USE AS SUBSTRATES AND BACK-UP FILTERS

Substrate mass gains have been found to be a source of very large errors when sampling industrial flue gases with inertial impactors in which glass fiber material is used for substrates. Ideally, the only mass change in a substrate should be that resulting from collection of particulate matter from the flue gas. However, if gas-phase reactions take place, involving components of the glass fiber substrate, then it is possible for substantial mass gains to occur, unrelated to the particle size distribution.

Previous Results

A report by Forest and Newman[2] indicates that mass gains in glass fiber filters are possible by conversion of SO_2 to various suflates.

The work of Charles Gelman and J. C. Marshall of the Gelman Instrument Company, makers of various filter media and equipment, seems to confirm that SO_2 absorption is the cause of the mass gains.[3] They acknowledge that a high pH glass fiber can absorb sulfur dioxide and thus cause erroneously high particulate weights. Pate and Lodge's work[4] using Na_2CO_3 treated glass filters as "dosimeters" for SO_2 exposure chambers, with mass gain of the filters being a time function of exposure to SO_2 was mentioned.

According to Gelman and Marshall, the SO_2 reaction on glass fiber could cause "a 30% error in the measurement of total suspended particulate matter" in an urban atmosphere. The new automotive catalytic mufflers could increase this error. It is possible that flue gases would give even higher errors, especially if the gases have a high moisture content, because the reactivity of SO_2 appears to increase at higher humidity.

Both quartz and glass fiber filter material were tested by Gelman. The quartz was found to be non-reactive to SO_2. The glass fiber materials, Type II and SpectroGrade, prepared with H_2SO_4, were low in SO_2 pickup. The SpectroGrade glass, prepared with HCl, picked up significant amounts of SO_2. (See Table IX.) Their explanation is that the glass prepared with H_2SO_4 has reacted to form $CaSO_4$ to prevent further reaction with SO_2 to form sulfates. The test used for SO_2 reactivity was to expose the filters to a water saturated atmosphere of SO_2 for 20 hours. Mass change of the filter was measured.

TABLE IX

SULFUR DIOXIDE PICKUP[3]

mg/Sheet - 20 Hour Exposure

	mg	Initial pH
SpectroGrade-HCl Siliconized	3	7.1
SpectroGrade HCl	17	9.4
SpectroGrade H_2SO_4	3	6.8
Type II Fiber H_2SO_4	3	6.8
Quartz	0	7.0
Quartz Alkali Strengthened	23 (est.)	9.5

Another type of SpectroGrade coated with an organic silicone resin showed low SO_2 pickup. This type of SpectroGrade with the silicone treatment is now standard type. Use of the siliconized SpectroGrade at elevated temperatures can result in the disappearance of the coating and SO_2 absorption by the filter medium since the filter is prepared with HCl.

Although the work by Gelman and Marshall showed quartz fiber media to be non-reactive with SO_2, the material has been found to be too fragile to be used successfully as an impactor substrate material.

Teflon is nearly inert and might be a good choice, although it cannot be used in hot electrostatic precipitators where flue temperatures regularly exceed 260°C (500°F). Another disadvantage of teflon is that particles tend not to adhere upon impaction, leading to scouring and particle bounce.

On the basis of strength, material integrity and particle retention, only glass fiber materials (or greases, used below 177°C [350°F])are left as suitable substrates, in spite of the problem posed by SO_2 uptake. These results imply that there is no inert filter material which is a usable substrate for cascade impactors as obtained directly from the manufacturers. Of the glass fiber materials tested, Whatman GF/A, GF/D, and Reeve Angel 934AH show the least mass gains when exposed to flue gases.

Preliminary Laboratory Screening Tests

The purpose of the laboratory screening tests was to gain an understanding of the mass changes that occur and to facilitate the selection of sufficiently inert filter material for impactor substrates. A suitable substrate material would be one which has stable low mass characteristics and is mechanically strong to resist cutting, tearing, and loss of material. Since the mass changes are apparently a result of chemical reactions involving the production of sulfates, the laboratory work was principally concerned with exposure of the substrate materials to sulfuric acid and/or SO_2 gas. The stability of mass changes over long time periods was investigated in order to evaluate the prospects for preconditioning techniques as a means for controlling mass changes. Two laboratory test methods were employed. One approach used a flow of gaseous SO_2 through the filter material, and the second involved soaking the material in hot sulfuric acid solution.

Exposure of Filter Media to SO_2

In this laboratory study glass fiber substrate materials were exposed to air, SO_2, and water vapor at an elevated temperature. Figure 10 shows a diagram of the conditioning apparatus. Dry air was preheated in the conditioning oven and then bubbled through a

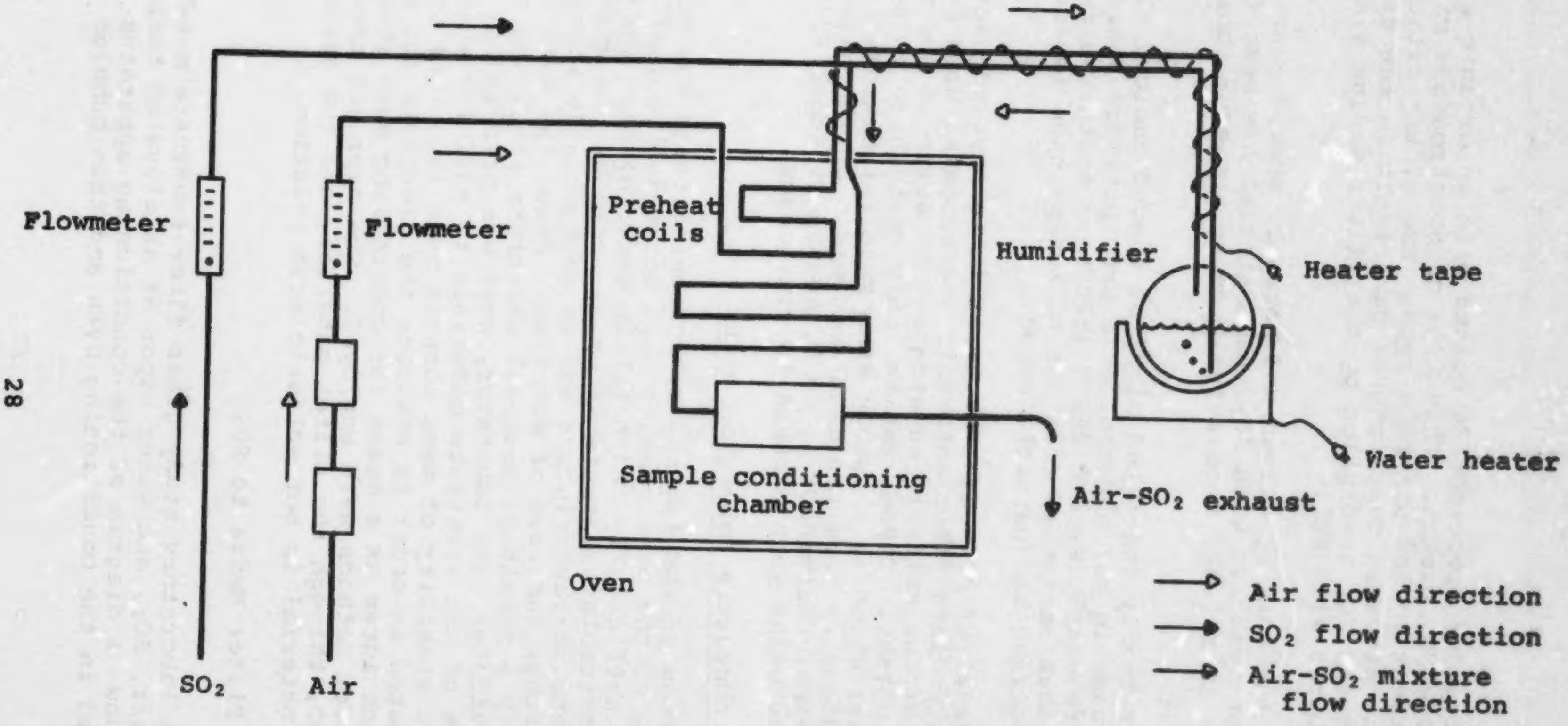

Flowmeter
Flowmeter
Preheat coils
Humidifier
Heater tape
Water heater
Sample conditioning chamber
Air-SO_2 exhaust
Oven
SO_2
Air
Air flow direction
SO_2 flow direction
Air-SO_2 mixture flow direction

Figure 10 Diagram of experimental set-up for filter substrate conditioning experiment

heated water container 60°C (140°F). Next, SO_2 was introduced to the heated and humidified air stream. All lines carrying SO_2 laden air was then passed through a chamber containing the filter media being tested. The chamber was designed so that conditioning gases flowed through the filter stack being conditioned.

Both gravimetric and pH determinations were used in investigating the rate of SO_2 uptake by the sample material. The procedure used to determine the filter pH was a modification of Gelman's method for 8" x 10" filter sheets.[3] Two 47 mm filters were used for each pH determination.

In one series of tests, four different kinds of glass fiber substrate materials were treated in the laboratory conditioning chamber on an hour-by-hour hasis. After each hour of conditioning the substrates were weighed, desiccated, reweighed, and the weights were recorded. It was found that desiccation resulted in no change in the weights, so this practice was discontinued. The four substrate materials tested were Reeve Angel 934AH, Gelman AE, Gelman SpectroGlass, and Whatman GF/A. All filters were 47 mm in diameter. Eight groups of twenty filters each were prepared and conditioned in the following order:

1. Reeve Angel 934AH
2. Gelman AE
3. Gelman SpectroGlass
4. Whatman GF/A
5. Reeve Angel 934AH
6. Gelman AE
7. Gelman SpectroGlass

Gas flow was such that the Reeve Angel material was exposed first. Figure 11 shows the results for the first nine hours of conditioning. The conditioning temperature was 260°C (500°F). Water saturated air with 5% SO_2 was pumped through the chamber at a rate of 2.1 lpm. Note that after nine hours of conditioning the Reeve Angel material had not gained but lost weight. However, the weight loss is miniscule and is probably due to handling. All others had gained significant amounts.

Sulfuric Acid Wash Treatment of Filter Media

Another approach to passivating impactor substrates was also investigated. Bundles of Reeve Angel 934AH and Gelman AE 47 mm filters were soaked in hot sulfuric acid-water mixtures for 90 minutes. These filters were then washed in distilled water, washed again in ethanol (ETOH) or isopropanol (IPA), dried, baked and desiccated. Upon conditioning for one hour under the conditions described above (260°C [500°F], air-water gas mixture with 5% SO_2) eighteen Gelman AE 47 mm filters gained 11.9 mg or 0.66mg/filter. Twenty untreated Gelman AE 47 mm filters gained 67.7 mg or 3.39 mg/filter with the same conditioning. Therefore, the

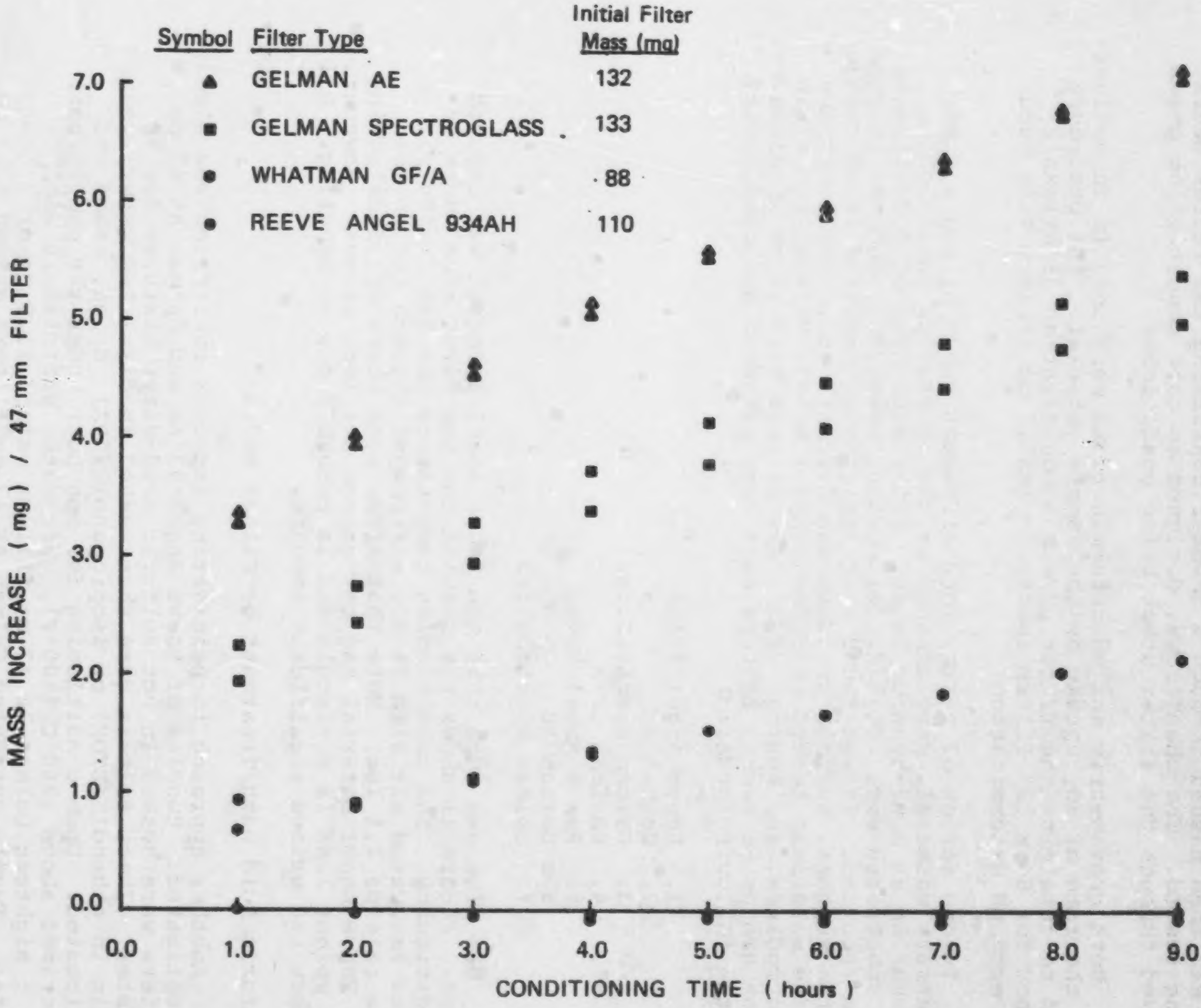


Figure 11. Mass gains of four types of glass fiber filter materials versus exposure time to a water-air-5% SO_2 gas mixture at 260°C (500°F).

sulfuric acid wash can make a difference when the filters are known to gain weight. The Reeve Angel material again showed no weight gains.

The hour-by-hour conditioning of the four different types of glass fiber filter substrate materials was continued, and mass gains were monitored for a total of 26 hours of conditioning. In addition, the sulfuric acid washed Gelman AE and Reeve Angel 934AH materials were laboratory conditioned on an hour-by-hour basis for a total of 18 hours. Figure 12 shows the mass gain per 47 mm filter versus laboratory conditioning time. Data for Gelman AE, AE acid washed, Gelman SpectroGlass, and Whatman GF/A are presented. Reeve Angel 934AH plain and acid washed filter materials were also conditioned, but since mass increase in this material was negligible these data were not graphed. The Gelman AE acid washed material gained approximately one third as much mass as the plain Gelman AE. Figure 12 also shows that even after 26 hours of laboratory conditioning mass gains may be expected with further conditioning.

Gas analyses were conducted on the conditioning gas at the inlet to the conditioning chamber. SO_2 and SO_3 concentrations were measured at approximately 10,000 ppm, and 3 to 5 ppm, respectively. Iron is a catalyst for the conversion of SO_2 to SO_3 at the conditioning temperature (22°C, 428°F). The conversion efficiency is small, less than one percent, but still enough SO_3 is produced to be detected. Since all the SO_2 carrying lines are stainless steel and since the conditioning chamber is stainless steel we should expect that the filters which have been SO_2 conditioned have also been exposed to SO_3.

Table X summarizes the end-point results presented in Figure 12. These data are presented in the order in which the 47 mm filters were conditioned in the stainless steel conditioning chamber (alundum filter holder). Results are presented on a mass gain per filter and percent mass gain basis.

In another series of tests, chemical analyses were made on the laboratory conditioned and unconditioned filters. Table XI shows the barium, calcium, and soluble sulfate concentration in two types of glass fiber filter material conditioned at SRI: Reeve Angel 934AH and Gelman AE. These 47 mm filters were analyzed when received, after being baked-out and desiccated and after being conditioned. The Reeve Angel material shows large amounts of calcium and miniscule amounts of barium and soluble sulfates, even after 12 hours of conditioning. The Gelman AE materials show large amounts of calcium as well, but after conditioning there is a great gain in soluble sulfates. This is reflected in the mass gains for this material. Each 47 mm filter gained on an average 2.93 mg. The initial pH of the Gelman AE material after baking was 9.8. With two hours of conditioning the pH dropped to 8.8. This is in contrast with the behavior of the Reeve Angel material. The pH of this substrate material stayed rather constant at about 5.9 to 6.7 before and after conditioning. Mass gains on conditioning for any length of time were very small.

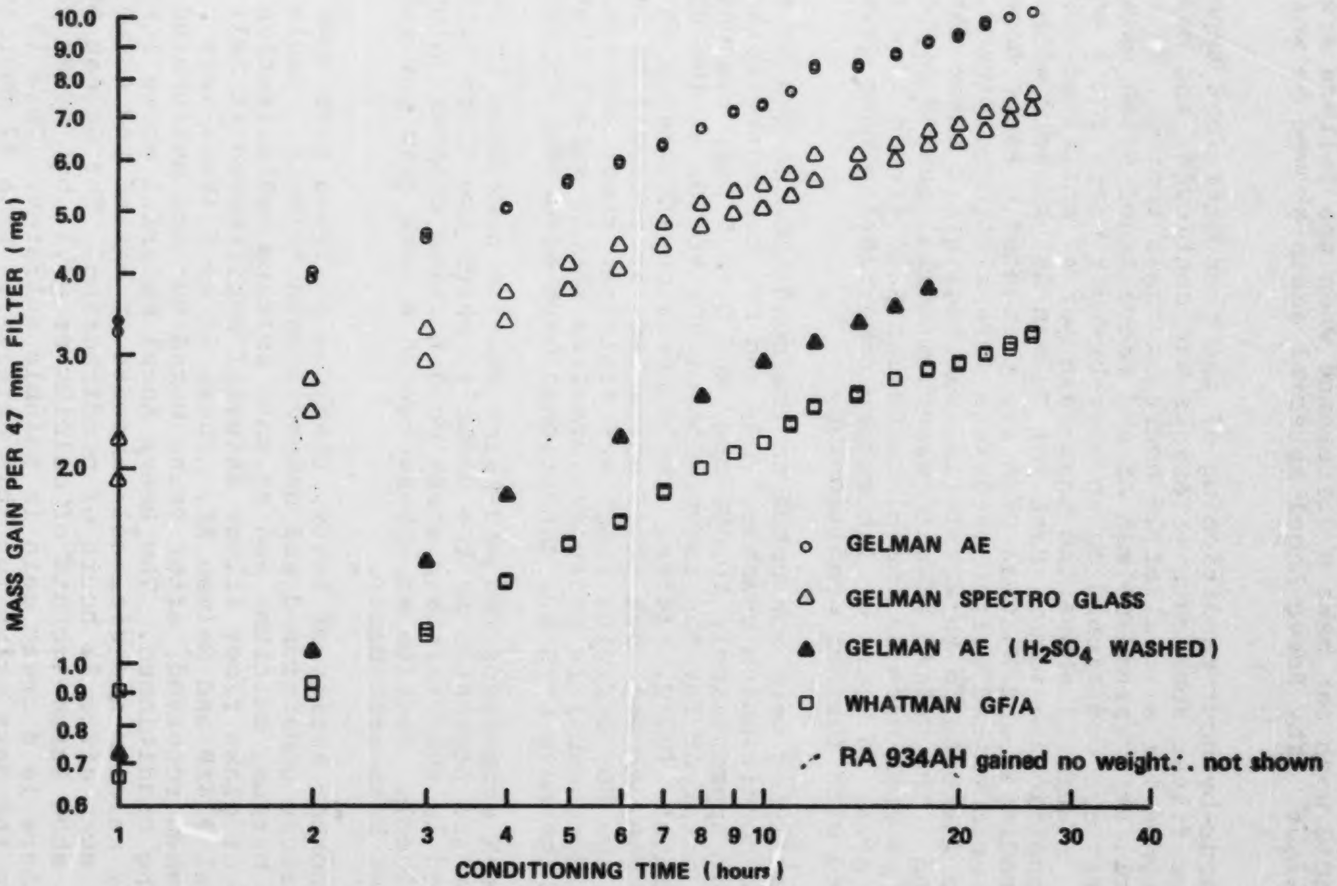


Figure 12. Mass increase per 47mm filter as a function of laboratory conditioning time.

TABLE X MASS GAINS OF 47 mm GLASS FIBER FILTER SUBSTRATE MATERIALS FROM LABORATORY CONDITIONING[1]

Material[2]	Batch Number	Number of 47 mm Filters Conditioned	Conditioning Time (Hours)	Mass Before Conditioning (grams)	Mass After Conditioning (grams)	Mass Gain per Filter (mg)	Percent Mass Gain
Reeve Angel 934AH	3307	20	26	2.1888	2.1881	-0.04	-0.03
Gelman AE	8204	20	26	2.6644	2.8735	10.46	7.84
Gelman Spectro Glass	8192-20232	20	26	2.6717	2.8160	7.22	5.40
Whatman GF/A	3563	20	26	1.7695	1.8349	3.27	3.70
Reeve Angel 934AH	3307	20	26	2.2149	2.2166	0.09	0.08
Gelman AE	8204	20	26	2.6266	2.8375	10.55	8.03
Gelman Spectro Glass	8192-20232	20	26	2.6522	2.8051	7.65	5.77
Whatman GF/A	3563	20	26	1.7361	1.8272	4.56	3.64
Reeve Angel 934 AH (Acid Washed)	4292	20	18	2.0968	2.0975	0.04	0.03
Gelman AE (Acid Washed)	8206	20	18	2.6939	2.7699	3.80	2.82

[1] 1% SO_2, 3-5 ppm SO_3, Saturated H_2O, 220°C (428°F)

[2] Filter materials are listed in order of conditioning in a stainless steel Alundum thimble holder.

TABLE XI

Barium, Calcium, and Soluble Sulfate Content in Two Glass Fiber Substrate Materials

Reeve Angel 934AH Substrate Material

	BARIUM		CALCIUM		Soluble SULFATE	
	Ba^{++} Mass (μg)	%BaO	Ca^{++} Mass (μg)	%CaO	SO_4^{--} Mass (μg)	%SO_4
Original	63	.06	14202	17.9	3.5	.003
After Bakeout	54	.05	14055	17.8	3.5	.003
After Conditioning (2 hours)	135	.13	14531	18.2	4.5	.004
After Conditioning (12 hours)	<10	<0.01	13820	17.6	92	.08

Gelman AE Substrate Material

	BARIUM		CALCIUM		Soluble SULFATE	
	Ba^{++} Mass (μg)	%BaO	Ca^{++} Mass (μg)	%CaO	SO_4^{--} Mass (μg)	%SO_4
Original	<10	<0.01	6094	6.5	<10	<0.0
After Bakeout	<10	<0.01	5470	6.1	<10	<0.0
After Conditioning (2 hours)	<10	<0.01	5758	5.9	3013	2.2

These results indicate that a laboratory induced sulfate mass gain can be made to occur in glass fiber filter materials. Whether or not this mass gain, or "conditioning" lasts is another question. To determine if the conditioning is a temporary effect, samples of these filters (16 to 20 filters per sample) were conditioned for 2 to 12 hours. Some were exposed to ambient air after conditioning, while others were desiccated.

Figures 13 and 14 show the results of these tests for the Reeve Angel 934AH material. Figure 13 shows the percent weight change versus days after conditioning for groups of filter conditioned for 2 hours and 12 hours. One group was exposed to ambient air after conditioning and another group was desiccated after conditioning. In both cases minute mass gains were seen for 12 hour conditioning and minute mass losses were seen for 2 hour conditioning. In either case there appears to be no reaction after conditioning resulting in an appreciable mass gain or loss. Figure 14 shows the pH of single filter samples measured after conditioning for 2 and 12 hours. As in Figure 13, one group was exposed to ambient laboratory conditions while another group was desiccated. This substrate material appears to have essentially no change of pH upon conditioning, and it is possible that since the material is only slightly acidic, changes in the pH of water used in the pH determination could have caused the changes shown in Figure 14. Whenever pH of a filter sample is measured, the pH of the water used is also measured. We believe this to be the case for the low pH recorded on day 2 of the desiccated sample and day 3 of the exposed sample. In this case the raw distilled water used in the pH determination had a measured pH of 4.32.

From these tests it would appear that, if pH is a good monitor, the conditioning has a lasting effect. Samples of this material, conditioned for 12 hours, which were stored under desiccation for as long as 77 days show no mass change and small change in pH (6.10 before, 6.77 after).

Field Studies

The effects of industrial flue gases on various filter substrate materials were studied in a series of field tests. Several types of filter material, listed in Table XII, were obtained from commercial suppliers for testing. In these experiments, unconditioned, laboratory conditioned, and acid washed glass fiber substrate materials were subjected to flue gases under various sampling conditions. Since the purpose of the evaluation is to determine procedures and identify materials which exhibit minimum weight changes for sampling applications, the filter media were exposed to flue gases for time intervals characteristic of an impactor run during stack sampling.

In the typical experimental procedure several filters are cut to size, if necessary, and mounted in 47 mm Gelman stainless steel

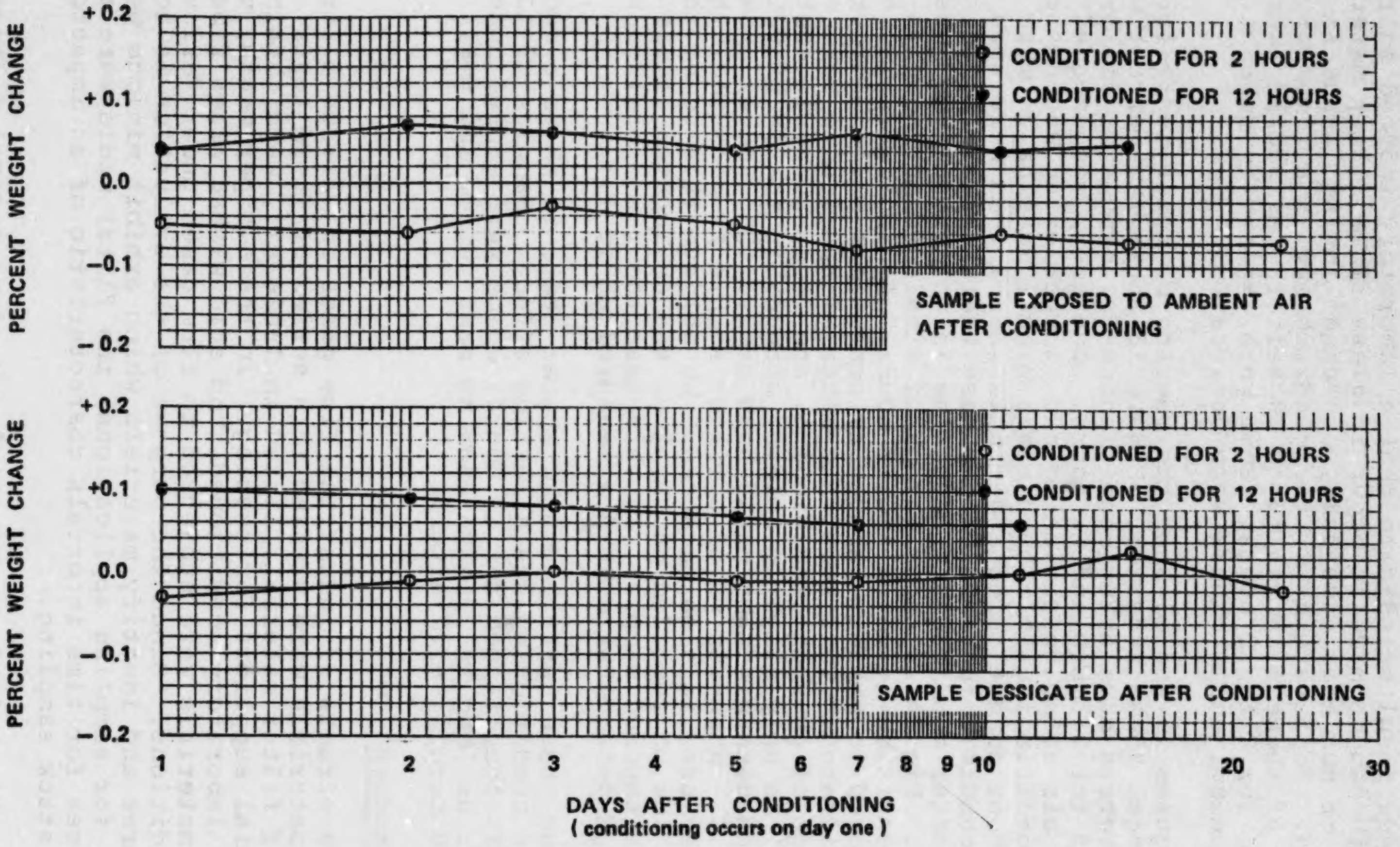


Figure 13 . Percent weight change for Reeve Angel 934AH glass fiber filter substrate material as a function of time after conditioning.

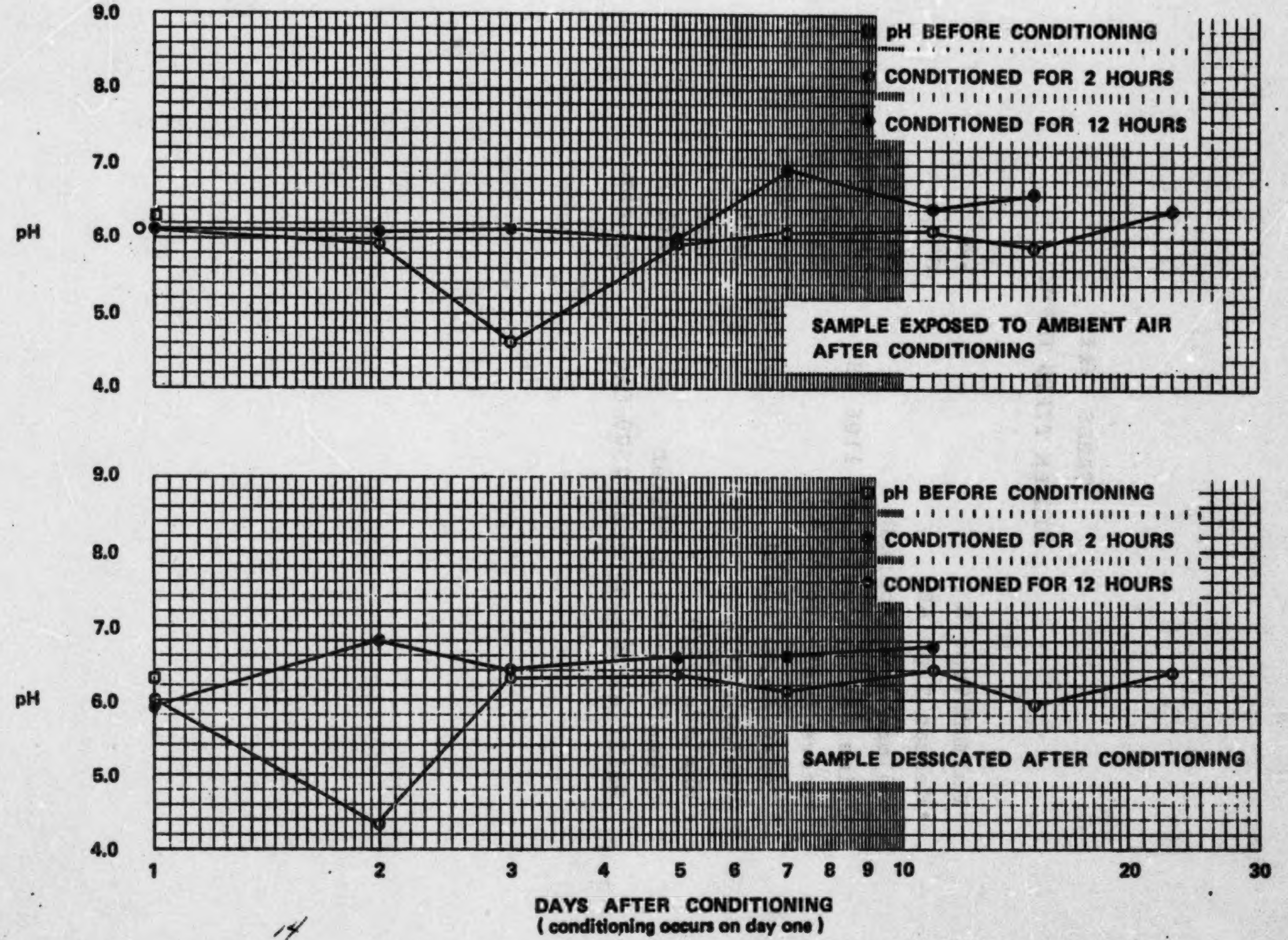


Figure 14. pH of Reeve Angel 934 AH glass fiber filter substrate material as a function of time after conditioning.

TABLE XII

FILTER TYPES TESTED

Gelman Type A	GA
Gelman Type AE	GAE
Gelman SpectroGrade	SA
Mine Safety Appliance 1106 BH	MSA 1106 BH
Reeve Angel 900AF	RA 900AF
Reeve Angel 934AH	RA 934AH
Whatman GF/A	GF/A
Whatman GF/D	GF/D
Chemplast Teflon Filter	Teflon
Pallflex Tissuquartz 2500 QAD	Quartz

filter holders. The filter holders were assembled as a series filter arrangement and run as an Andersen Stack Sampler would be run. The first filter holder was a pre-filter which removed the particulate material. The remainder of the filters, each in its holder, were exposed only to the flue gas. Isokinetic sampling was not considered important in order to approximate the exposure of the substrates to flue gases in typical sampling situations. In some cases, impactors loaded with substrates in the usual fashion were used with prefilters rather than using the series of 47 mm filters described above.

The field test sites included ESP applications at a gas fired cement kiln and six different coal fired steam plants, covering a temperature range from about 100 to 350°C (212 to 662°F).

The results of tests made at two steam plants, A and B, are especially interesting. The substrates were supplied by Andersen 2000, Inc. At steam plant A the gas temperature was 149°C (300°F) and at steam plant B the gas temperature was 316°C (600°F). Determinations of soluble sulfates were made on the substrates after each run. The results of those suflate determinations are plotted against the gain in mass per substrate in Figure 15. The solid line indicates a perfect one-to-one correspondence between the observed mass gains and amount of soluble sulfates found on each filter. These data fall very close to this line of perfect agreement. No correlation was found between flue gas SO_2 concentration and blank mass gains in this test series. Data from two tests at Plant B are shown in Table XIII. These data show a similar near perfect agreement between the weight increases determined gravimetrically and the chemically determined sulfate content on the second test. No weight increases were observed during the first test. When queried as to whether they had changed suppliers for their substrate material in the time between the first and second tests at steam plant B, Andersen 2000, Inc. replied that they had used and were using Gelman Tyep A glass fiber filter substrate material but that Gelman had changed their manufacturing process so that the Type A material used in the second plant B test was washed in hydrochloric acid whereas the material used in the first test at plant B was made with a sulfuric acid wash.

Effects of Flue Gas Temperature

Figure 16 shows the relation between substrate mass gain and flue gas temperature. With the exception of the "pre 6/74" point, a linear relationship seems to hold. The "pre 6/74" represents data from the first of the two tests conducted at steam plant B where the substrate was quite neutral compared to the basic substrates in the second test.

Figure 17 shows the results of tests of several types of 47 mm glass fiber filter substrate material at different flue gas conditions and temperatures. This again illustrates the apparent relationship between flue gas temperature and substrate mass gain as well as the insensitivity of SO_2 concentration.

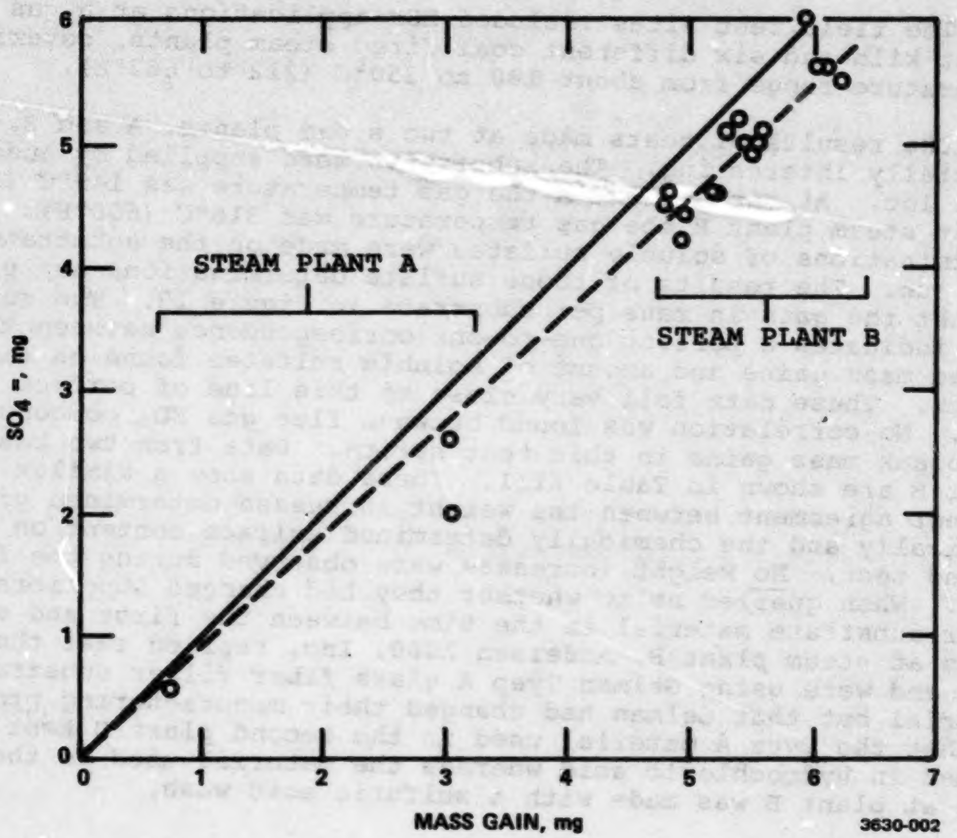


Figure 15. Comparison of mass of sulfate on blank Andersen impactor substrates and observed anomalous mass increases.

TABLE XIII

SOLUBLE SULFATE ANALYSES OF FILTERS USED AT STEAM PLANT B

Unused samples from batch used at steam plant B (6/74). No samples from actual blank runs (where no mass gains were observed) are available.

Sample No.	pH[a]	Total mg[b] $SO_4^{=}$/filter	Reported wt gain, mg
11	6.6	∼0.2	–
18	6.8	∼0.2	–
1F	6.9	∼0.2	
Set 1 from second steam plant B test (1/75)			
Unused, perforated, unbaked	9.4	∼0.2	–
Unused, perforated, baked	9.4	∼0.2	–
S11	7.6	4.4	4.98
S12	7.7	4.2	4.96
S13	7.4	5.1	5.58
S14	7.0	5.0	5.56
S15	7.1	5.6	6.06
S16	7.1	5.6	6.10
S17	7.0	6.0	5.94
S18	6.8	5.6	6.04
Set 2 from second steam plant B test (1/75)			
Unused, perforated, baked	9.3	∼0.2	–
Unused, solid, baked	9.7	∼0.3	–
S20	6.8	7.3	8.34
S21	6.7	5.1	5.30
S22	7.1	4.6	5.22
S23	7.4	5.2	5.40
S24	7.2	4.5	4.80
S25	7.3	5.0	5.44
S26	7.2	4.6	5.24
S27	7.1	4.9	5.50
S28	7.0	4.6	4.86
S2F	8.2	5.5	6.24

---

a. pH determined after the filter sample was in contact with 10 ml of distilled water (pH 5.6) for 1 hr.

b. The total soluble sulfate was determined by a $Ba(ClO_4)_2$ titration following a water extraction of the sample.

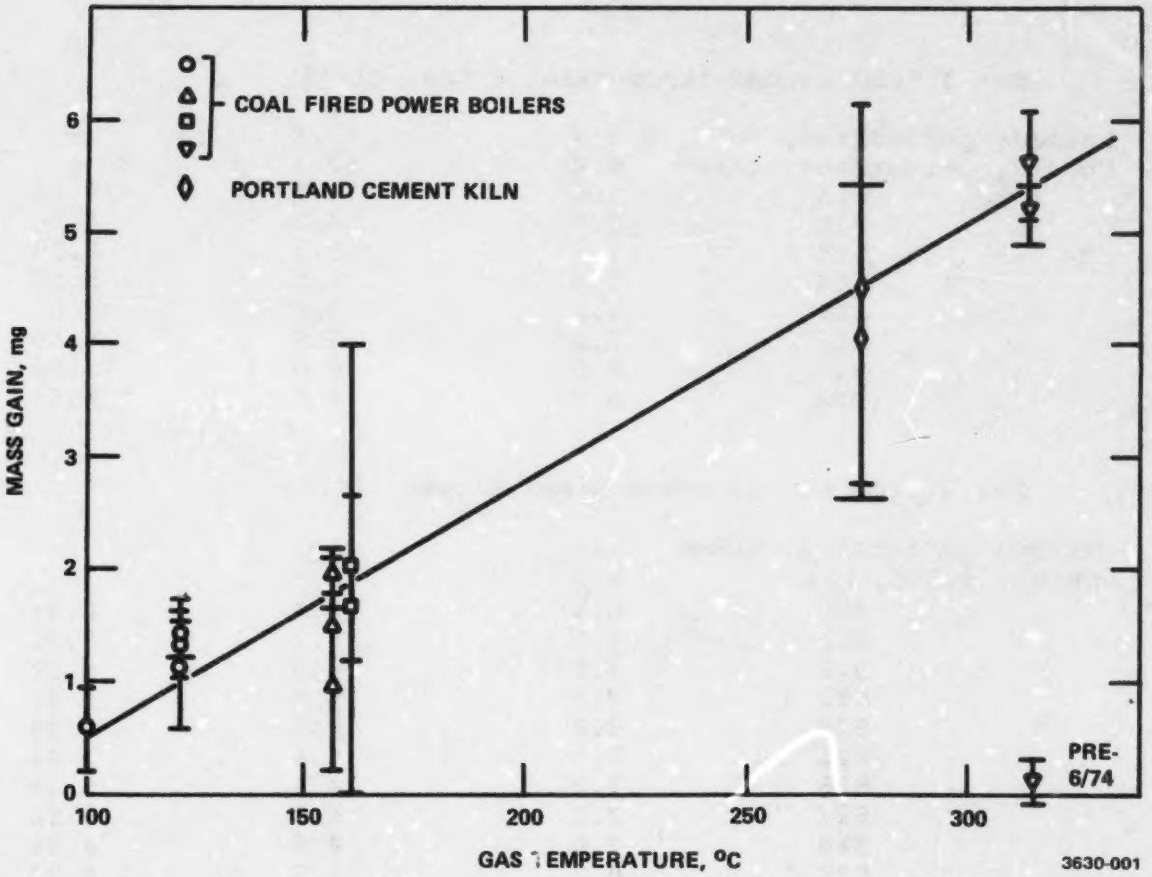


Figure 16. Anomalous mass increases of Andersen glass fiber impaction substrates at different flue gas temperatures.

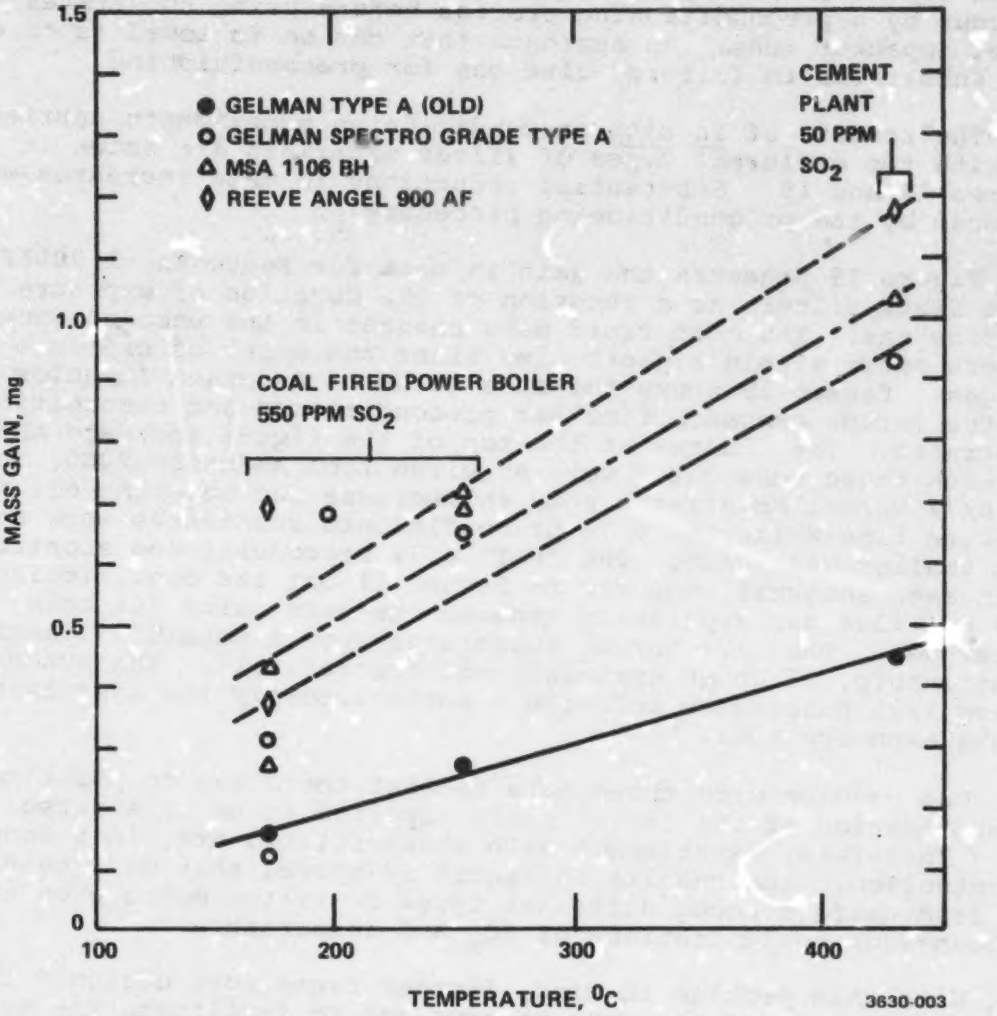


Figure 17. Anomalous mass gains of various 47mm diameter glass fiber filters at different temperatures. (60 minute samples at flowrates of 0.25 ACFM)

In situ Preconditioning

Because mass gains in filter substrate media are related to chemical reactions occurring as a result of the action of hot flue gas on filter components, it may be possible to force the reactions to occur by a preconditioning process before using substrates in actual impactor runs. An approach that can be followed is to expose substrates to filtered flue gas for preconditioning.

The results of *in situ* preconditioning experiments carried out with two different types of filter materials are shown in Figures 18 and 19. Substantial reductions in mass increases were produced by the preconditioning procedure.

Figure 18 presents the gain in mass for Reeve Angel 900AF glass fiber filters as a function of the duration of exposure to the flue gas. The most rapid mass changes in the unconditioned filters occur within a short time after the onset of exposure to the gas. Figure 19 shows the mass gains of Andersen Impactor substrates versus exposure time for preconditioned and unconditioned substrates. The dates at the top of the figure indicate the time at which these substrates were acquired from Andersen 2000, Inc. The 6/74 Normal Substrates show an increase and leveling off with exposure time while the 6/74 preconditioned substrates show somewhat smaller mass gain. The "HOT" 6/74 preconditioned substrates would seem abnormal compared to Figure 11 but the conditioning with hot flue gas apparently reduced the mass gains for this filter set. The 1/74 Normal Substrates show a possible linear relationship, although certainly not conclusively. The preconditioned 1/75 Substrates indicate a satisfactorily low mass gain versus exposure time.

One problem with these data is that there was no positive identification of the filter media supplied to us by Andersen 2000, Inc. Therefore, experiments with these filters are, in a sense, uncontrolled. The results in Figure 17 showed that mass gains are far from uniform among different types of filter media when exposed to identical concentrations of SO_x and temperature.

With this problem in mind, further tests were designed to gain an understanding of the mass changes and to facilitate the selection of a sufficiently inert filter material for impactor substrates. A suitable substrate material would be one which has stable mass characteristics and is mechanically strong to resist cutting, tearing, and loss of material.

Several types of filter material were obtained from commercial suppliers for testing: Gelman Types A, AE, and SpectroGrass glass fiber filter material, Mine Safety Appliance 1106-BH glass fiber filter material; Reeve Angel 900AF and 934AH glass fiber material; Whatman GF/A and GF/D glass fiber material; Chemplast teflon filter membranes; and Pallflex Tissuquartz 2500 QAD (see Table XII). The

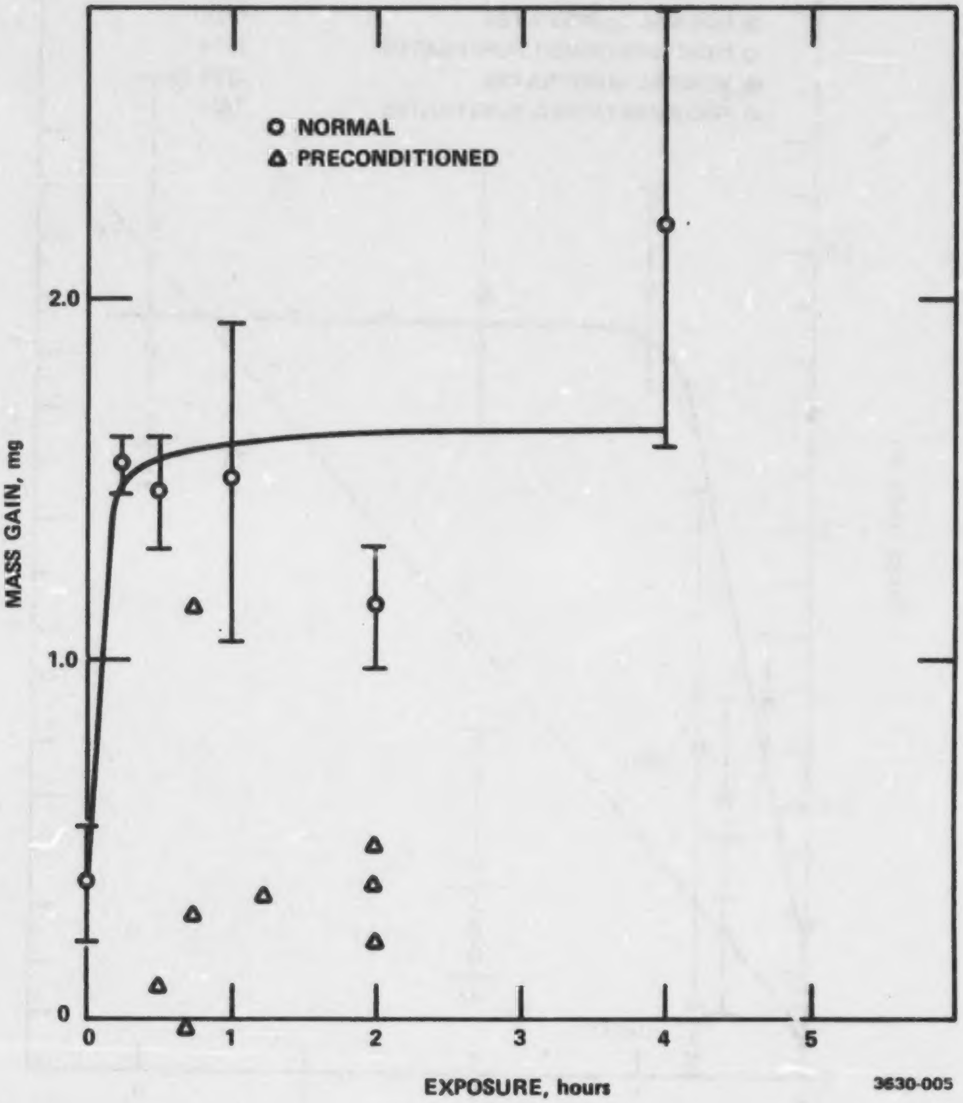


Figure 18. Anomalous mass gain of 64mm diameter Reeve Angel 900AF glass fiber filters.

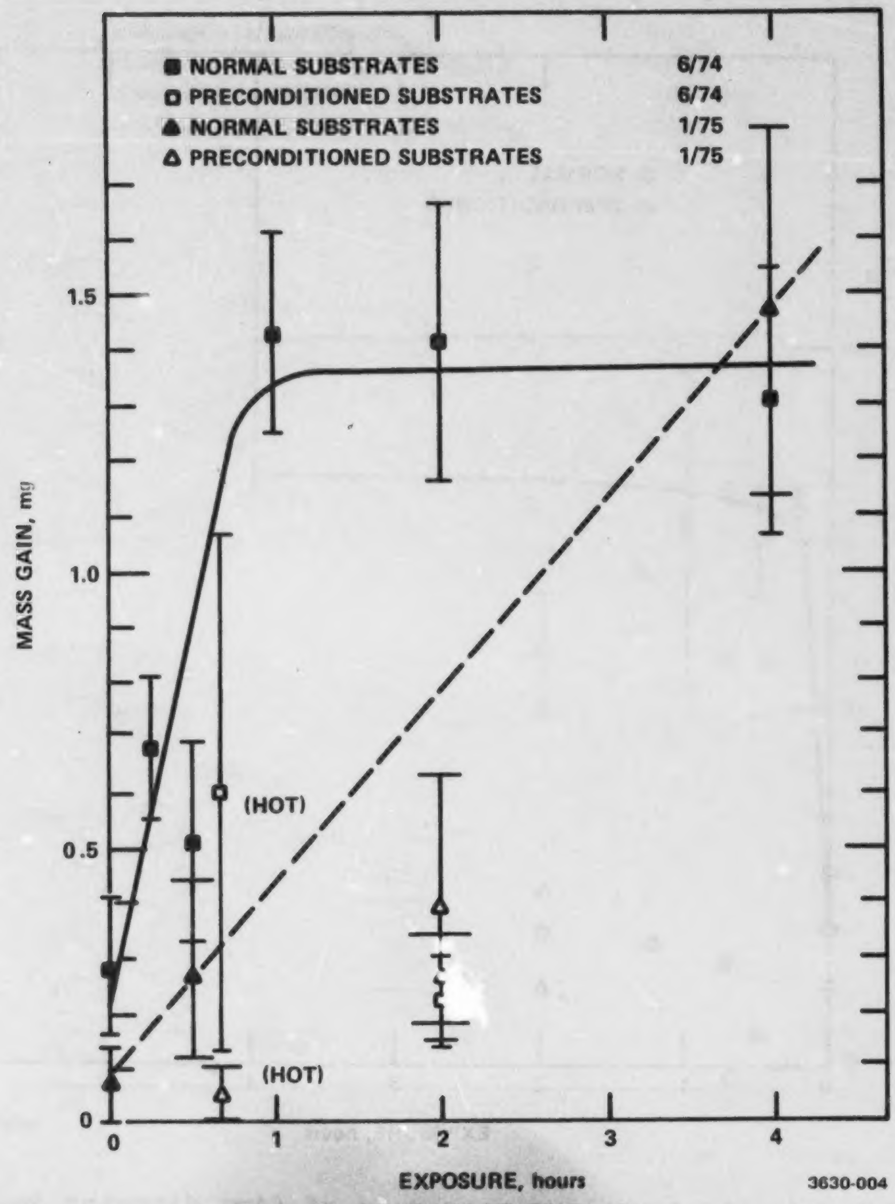


Figure 19. Anomalous mass gains of Andersen impactor glass fiber impaction substrates.

method chosed to test the filter media was to expose them directly to the flue gases for time intervals characteristic of an impactor run. Several Gelman stainless steel 47 mm filter holders were used to accomplish this. These holders were assembled as a series filter arrangement and run just as an Andersen Stack Sampler would be run. The first filter holder was a pre-filter which removed the particulate material. The remainder of the filters, each in its holder, were exposed only to the flue gas.

Before the weighed filter samples were loaded in the Gelman holders, they were cut to 47 mm diameter where necessary, baked in a laboratory oven at 287°C (550°F) for two to three hours, and desiccated for at least twenty-four hours. The samples remained in the desiccator until just prior to use.

Two sampling sites were chosen for testing of the filter media. Both sources were placed where previous impactor runs had been made, and the sources were of different types. The first testing was done at the outlet of a hot-side ESP at a cement plant between February 12th and February 21st, 1975. Two gas fired kilns were in operation while the tests were being performed. Outlet temperatures were in the neighborhood of 260°C (500°F). Other data, including flow rates and mass gains, are listed in Table XIV. Filter 1a includes the particulate catch in each case.

The second site was a power plant (steam plant A). Here tests were performed at the coal-fired boiler precipitator outlet during two periods. The first period was February 25 to February 28, 1975. Flue gas temperatures ranged from 130°C (275°F) to 180°C (335°F). Other data are listed in Table XV. The second testing at steam plant A was April 1 to April 3, 1975, with flue temperatures varying from 138°C (280°F) to 174°C (345°F). Table XVI contains these data.

In the test at the cement plant and the first test at steam plant A, there was a problem with the filter material sticking to the O-ring and the support screen of the holder. This created a nuisance, and also added the possibility that some material might be overlooked. For the tests in April at steam plant A, teflon gaskets were cut which fitted directly under the O-ring and on the support screen. The filter sample was held between teflon gaskets and the gaskets were preweighed with the filter sample.

Two identical 47 mm filters were run in each holder. Originally, it was hoped that we might be able to obtain mass gain data from each of these, but because of a tendency of the filters to adhere to one another, the mass gains of both were lumped together. All the results are shown in Table XVI.

The sampling times and flow rates used were supposed to approximate the sampling time and flow rate of a typical Andersen Stack Sampler test. Sampling times varied from thirty minutes to eight

TABLE XIV

CEMENT PLANT - SUBSTRATE MASS GAINS

	Run Number		CC-1			CC-2			CC-3			CC-4	
	Date		2-12-75			2-18-75			2-19-75			2-21-75	
("Hg)	Amb. Pres.		29.7			29.5			29.7			29.9	
(°F)	Amb. Temp		62			68			48			58	
(°F)	Stack Temp		510			480			480			505	
(ft³)	Gas Vol.		12.287			23.808			24.030			33.068	
(°F)	Avg. Gas Meter Temp		58			74			53			79	
(min)	Run Time Ori. ID		60 3348-.059			120 3348-.059			120 3348-.059			240 3348-.059	
(ml)	Cond. H_2O % H_2O		74 (29.6)	22% used for flowrate		121 (26.6)	22.0		131 (27.2)	22.0		97 (28.0)	22.0
(acfm)	Flow Rate: - Ori. - Gas Meter		0.414 0.438	} 0.426		0.383 0.390	} 0.387		0.391 0.410	} 0.400		0.147 0.1535	} 0.150
("Hg)	Avg. Probe ΔP		2.4			3.0			3.0			15.0	
("H_2O)	Ori. ΔP		11.0			10.3			10.4			2.6	
SUBSTRATE MASS GAINS (mg)	1a	GA	3.22 mg		GA	10.58 mg		GA	9.48 mg		GA	4.38	
	1b	-	-	Silicone o-rings stuck to filters	GA	0.44		GA	0.84		MSA 1106 BH	2.14	
	2	SA	0.28		SA	0.92	Slight brown ring	MSA 1106 BH	1.42		Teflon	-0.02	
	3	GA	-0.48		RA 900 AF	1.16		RA 900 AF	1.70		GA	0.90	
	4	-	-		MSA 1106 BH	1.02	Slight brown ring	SA	1.66	Stuck to metal support	RA 900 AF	2.40	
	5	-	-		-	-					MSA 1106 BH	1.92	
	6	-	-		-	-					Teflon	0.00	
						Teflon o-rings			Teflon o-rings			Teflon o-rings	

hours, which met the above requirement. The flow rates used, however, were somewhat below that of the ideal Andersen Flow Rate of 0.5 cfm. On all of the tests in which the teflon membranes were used, the flow rates were only half as high as the desired flow rate of 0.5 cfm because the pressure drop across the membrane was large. (Even so, the ratio between flow rate and filter surface area at these lower flow rates approximated the same ratio of these quantities for an Andersen Stack Sampler.) No attempt was made at isokinetic sampling since only the flue gas was of interest. After each run, the filters were desiccated for at least 24 hours and weighed. Any gas-phase reaction was then detected by a mass change.

The results of all the tests are listed in the tables. From these results, no definite trend emerged to indicate that the mass gains depended upon flow rate. However, there seems to be a relation between mass gain and total exposure time of the filter to the flue gas, regardless of the flow rate.

Both Whatman types GF/A and GF/D, and the Reeve Angel 934AH showed little tendency for mass gain. The Pallflex quartz showed loss of mass, but it was very fragile and tended to break and tear easily, which may have resulted in a loss of some of the filter material.

Chemical analyses were performed on all the filter materials that had been tested in the Gelman holders. This includes the tests at the cement plant and those at steam plant A. Soluble sulfate determinations and pH tests were run on each sample except the teflon samples, which had shown very little mass change. These results are shown in Tables XVII, XVIII, and XIX.

The data in these tables indicate that sulfate is responsible for the majority of the observed mass gain on each filter. (Some filters which had small mass gains appeared to have picked up little sulfate, but this may only reflect the limits of accuracy in the determination of sulfate.) In each case, the pH of the sample was more acidic after it had been run, indicating possible sulfate gain.

The results of these tests can be summarized as follows:

> The pH of the filters varied widely from batch to batch before testing.
>
> There was a definite correlation between high initial pH and mass gains upon testing.
>
> The pH decreased during testing.
>
> A large fraction of the mass gain in every case was found to be the result of sulfate formation on the filter media.

TABLE XV

STEAM PLANT A

	Run Number	BRSP-1	BRSP-2	BRSP-3	BRSP-4	BRSP-5	BRSP-6	BRSP-7
	Date	2-25-75	2-25-75	2-26-75	2-26-75	2-27-75	2-28-75	2-28-75
("Hg)	Amb. Pres.	29.08	29.08	29.44	29.42	29.36	29.10	29.10
(°F)	Amb. Temp.	54	61	53	69	64	41	50
(°F)	Stack Temp.	275	265	275	275	300	355	355
(ft³)	Gas Vol.	11.062	18.767	80.928	34.269	140.245	8.433	-
(°F)	Avg. Gas Meter Temp.	81	73	81	82	79	74	-
(min)	Run Time Ori. ID	60 3348-.059	60 3348-.059	240 3348-.059	120 3348-.059	480 3348-.059	30 3348-.059	0 -
(ml)	Cond. H_2O % H_2O	6.0 (3.8) 7.5 used in flow-rate calculations	11.4 (4.9) 7.5	57.5 (5.6) 7.5	23.8 (5.7) 7.5	90.8 (5.7) 7.5	4.0 (4.6) 7.5	- -
(acfm)	Flow Rate: -Ori. -Gas Meter	0.183 0.189 0.186	0.249 0.258 0.254	0.286 0.285 0.286	0.231 0.239 0.235	0.230 0.235 0.232	0.266 0.269 0.268	- -
("Hg)	Avg. Probe ΔP (+ ΔP across orifice)	8.8	13.65	12.6	12.7	13.95	12.5	-
("H_2O)	Ori. ΔP	6.5	14.9	10.5	12.8	12.7	12.9	-
SUBSTRATE MASS GAINS (mg)	1a	GA 12.29 mg	GA 26.50	GA -	GA 37.99	GA -	GA 23.41	GA 0.34
	1b	MSA 1106 BH 0.43	GA 0.44	RA 900 AF 0.73	MSA 1106 BH 0.88	GA 3.27 Moderately brown on edge.	MSA 1106 BH 0.69	GA 0.38
	2	GA 0.16	RA 900 AF 0.69	MSA 0.16	RA 900 AF 0.83	MSA 1106 BH 3.65 Moderately brown near o-ring	SA -0.12 Torn	MSA 1106 BH 0.77
	3	RA 900 AF 0.37	MSA 1106 BH -0.14 Stuck to o-ring & support severely	Teflon 0.01	GA 0.24	SA 0.68 Severely cut light brown	GA 0.27	SA -0.10 Torn
	4	Teflon 0.01	Teflon 0.00	SA 0.12	SA 0.18	GA 0.53 No discoloration	SA 0.66	GA 0.38
	5	MSA 1106 BH 0.27	RA 900 AF 0.56	GA -0.04 Severely cut	GA 0.61	Teflon 0.00	Teflon -0.01	MSA 1106 BH 0.70
	6	GA 0.19	GA -0.74 Stuck to o-ring & cut by it.	SA 0.31	Teflon 0.07	MSA 1106 BH 1.00 Slight browning	MSA 1106 BH 0.71	Teflon 0.06
			All filters stuck slightly to support. As much as possible recovered.	This (and all that follow) are Spectro Grade A from batch #192.				This set was not run: placed in stack and taken out immediately.

TABLE XVI

STEAM PLANT A

	Run Number	BRSP-8	BRSP-9	BRSP-10	BRSP-11	BRSP-12	BRSP-13
	Date	4/1/75	4/2/75	4/2/75	4/2/75	4/3/75	4/3/75
("Hg)	Amb. Pres.	29.28	29.15	29.15	29.15	29.12	29.12
(°F)	Amb. Temp.	84	78	92	89	48	57
(°F)	Stack Temp.	315	315	315	315	345	280
(ft^3)	Gas Vol.	52.172	13.004	6.531	6.516	50.284	12.697
(°F)	Avg. Gas Meter Temp.	103	92	107	99	53.5	69
(min.)	Run Time	240	60	30	30	240	60
(ml.)	Cond. H_2O	48.9	11.0	6.0	5.8	48.8	13.3
	% H_2O	(6.6) 7.5 used in flow-rate calculations	(5.2) 7.5	(5.9) 7.5	(5.6) 7.5	(6.1) 7.5	(6.2) 7.5
	Ori. ID	3348 - .059	3348 - .059	3348 - .059	3348- .059	3348 - .059	3348- .059
("H_2O)	Ori. ΔP	9.7	10.3	10.3	10.3	10.3	10.3
("Hg)	Avg. Probe ΔP	7.1	4.35	4.75	4.65	6.6	4.35
(acfm)	Flowrate:						
	- Ori.	0.242	0.267	0.261	0.262	0.272	0.260
	- Gas meter	0.238	0.272	0.262	0.266	0.266	0.265
SUBSTRATE MASS GAINS (mg)	* 1a	GA -	GA -	GA -	GA -	GA -	GA -
	1b	GA 3.68	GA 1.74	GA 1.00	GA 0.84	GA 2.74	GA 0.12
	2a	GAE 8.54	GAE 1.26	GAE 1.76	GAE 0.96	GAE 6.66	GAE 1.08
	2b	GAE	GAE	GAE	GAE	GAE	GAE
	3a	SA 2.02	SA 0.00	SA 0.08	SA -0.12	SA 4.06	SA 0.10
	3b	SA	SA	SA	SA	SA	SA
	4a	Quartz -0.50	Quartz -1.96	Quartz 0.12	Quartz -1.40	Quartz -5.16	Quartz 0.66
	4b	Quartz	Quartz	Quartz	Quartz	Quartz	Quartz
	5a	GF/A 0.52	GF/A 0.02	GF/A 0.08	GF/D 0.00	GF/D -0.04	GF/D 0.18
	5b	GF/A	GF/A	GF/A	GF/D	GF/D	GF/D
	6a	RA 934 AH 0.46	RA 934 AH 0.02	RA 934 AH 0.24	RA 934 AH 0.04	RA 934 AH 0.08	RA 934 AH 0.32
	6b	RA 934 AH	RA 934 AH	RA 934 AH	RA 934 AH	RA 934 AH	RA 934 AH

*Combined weight of the two filters per holder, except for holder 1 where the weight of the prefilter (1a) has been eliminated.

NOTE:

GA - Gelman Type A
GAE - Gelman Type AE
SA - Gelman Spectro Glass Fiber, Type A
Quartz - Pallflex Tissuquartz 2500 QAD
GF/A - Whatman GF/A
GF/D - Whatman GF/D
RA 934 AH - Reeve Angel 934 AH

TABLE XVII

FILTER BLANKS (UNTREATED)

Type	mg $SO_4^{=}$	pH
GA	0.04	8.6
MSA	0.18	9.3
RA 900 AF	0.06	9.8
SA	Negligible	5.6
Quartz	Negligible	6.6
GAE	Negligible	9.2
RA 934 AH	0.06	7.2
GF/A	Negligible	8.1
GF/D	Negligible	7.0

TABLE XVIII

CEMENT PLANT

Run Number		CC-1					CC-2					CC-3			
		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed*		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed
1a	GA	3.22	2.1	7.1	½	GA	10.58	5.02	8.1	½	GA	9.48	4.83	7.7	½
1b	-	-	-	-	-	GA	0.44	0.10	8.0	Whole	GA	0.84	0.27	7.2	½
2	SA	0.28	0.44	8.1	Whole	SA	0.92 Slight brown ring	0.94	8.1	½	MSA 1106 BH	1.42	0.90	7.7	½
3	GA	-0.48	-		-	RA 900 AF	1.16	0.83	7.7	½	RA 900 AF	1.70	1.45	8.1	½
4	-	-	-	-	-	MSA 1106 BH	1.02 Slight brown ring	0.76	7.7	½	SA	1.66 Stuck to metal support	1.85	8.7	½
5	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
6	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
			Silicone o-rings stuck to filters				Teflon o-rings					Teflon o-rings			

*On those filters containing relatively large amounts of particulate, only a portion of the filter was analyzed. Sulfate was calculated on a whole filter basis.

TABLE XVIII
(CONTINUED)

CC-4

	Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed
GA	4.38	2.0	7.4	¼
MSA 1106 BH	2.14	1.67	6.9	¼
Teflon	-0.02	-	-	-
GA	0.90	0.30	7.1	½
RA 900 AF	2.40	2.27	7.0	¼
MSA 1106 BH	1.92	1.52	7.5	¼
Teflon	0.00 Teflon o-rings	-	-	-

TABLE XIX

STEAM PLANT A

Run Number	BRSP-1					BRSP-2				
		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed*		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed
1a	GA	12.29	0.56	5.6	¼	GA	26.50	0.91	5.4	¼
1b	MSA 1106 BH	0.43	0.17	5.7	Whole	GA	0.44	0.04	5.9	Whole
2	GA	0.16	0.06	5.5	Whole	RA 900 AF	0.69	0.20	6.9	Whole
3	RA 900 AF	0.37	0.26	5.6	Whole	MSA 1106 BH	-0.14	Stuck to o-ring and support	-	
4	Teflon	0.01				Teflon	0.00			
5	MSA 1106 BH	0.27	0.11	6.7	Whole	RA 900 AF	0.56	0.26	7.7	Whole
6	GA	0.19	0.04	6.4	Whole	GA	-0.74	Stuck	-	
						All filters stuck at least slightly				

*Same note as Citadel

TABLE XIX
(CONTINUED)

Run Number		BRSP-3					BRSP-4			
		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed
1a	GA	-	-	-		GA	37.99	-	4.4	¼
1b	RA 900 AF	0.77	0.22	4.2	Whole	MSA	0.88	0.26	5.5	Whole
2	MSA	0.16	0.11.	4.5	Whole	RA 900 AF	0.83	0.25	5.7	Whole
3	Teflon	0.01	0.00	5.8	Whole	GA	0.24	0.06	5.1	Whole
4	SA	0.12	0.23	4.8	Whole	SA	0.18	0.29	5.3	Whole
5	GA	-0.04	Severely cut	-	-	GA	0.41	0.03	5.1	3/4
6	SA	0.31	0.05	4.8	Whole	Teflon	0.07			

TABLE XIX
(CONTINUED)

Run Number		BRSP-5					BRSP-6				
		Mass Gain (mg)	Sulfate (mg)		pH	Filter Portion Analyzed		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed
1a	GA	-	-		-	-	GA	23.41	1.28	3.9	¼
1b	GA	3.27	1.95	Moderately brown on edge	3.0	¼	MSA 1106 BH	0.69	0.24	6.4	Whole
2	MSA 1106 BH	3.65	2.34	Moderately brown near o-ring	3.0	¼	SA	-0.12	Torn	-	-
3	SA	0.68	1.40	Severely cut	3.0	Whole	GA	0.27	0.02	6.5	Whole
4	GA	0.53	0.103		5.6	Whole	SA	0.66	0.10	5.7	Whole
5	Teflon	0.00	-		-	-	Teflon	-0.01	-	-	-
6	MSA 1106 BH	1.00	0.52	Slight brown ring	5.8	¼	MSA 1106 BH	0.71	0.28	6.4	Whole

TABLE XIX
(CONTINUED)

BRSP-7

Run Number		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed
1a	GA	0.34	0.12	6.8	Whole
1b	GA	0.38	0.12	6.9	Whole
2	MSA 1106 BH	0.77	0.37	7.8	Whole
3	SA	-0.10	Torn	-	-
4	GA	0.38	0.08	7.2	Whole
5	MSA 1106 BH	0.70	0.31	7.8	Whole
6	Teflon	0.06	-	-	-

This set not run; placed in stack, heated for 30 minutes, and taken out.

BRSP-8

Run Number		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed
1b	GA	3.68	1.62	3.4	½
2a & b	GAE	8.54	5.16	3.3	½ each filter
3a & b	GSA	2.02	1.54	3.5	½ each filter
4a & b	Quartz	-0.50	Negligible	3.6	½ each filter
5a & b	GF/A	0.52	Negligible	5.9	2 whole filters
5a & b	GF/D	-	-	-	-
6a & b	RA 934	0.24	Negligible	5.6	2 whole filters

TABLE XIX
(CONTINUED)

Run Number	BRSP-9	Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed	BRSP-10	Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed
1b	GA	1.74	0.92	3.4	½	GA	1.00	0.42	3.7	Whole
2a & b	GAE	1.26	0.46	5.8	½ each filter	GAE	1.76	0.29	7.2	½ each filter
3a & b	GSA	0.00	0.05	5.3	2 whole filters	GSA	0.08	0.04	5.9	2 whole filters
4a & b	Quartz	-1.96	-	-	-	Quartz	0.12	Negligible	3.5	2 whole filters
5a & b	GF/A	0.02	Negligible	6.6	2 whole filters	GF/A	0.08	-	-	-
5a & b	GF/D	-	-	-	-	GF/D	-	-	-	-
6a & b	RA 934 AH	0.02	Negligible	6.1	2 whole filters	RA 934 AH	0.24	Negligible	6.3	2 whole filters

TABLE XIX
(CONTINUED)

Run Number		BRSP-11				BRSP-12				
		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed		Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed
1b	GA	0.84	0.37	4.1	Whole	GA	2.74	1.48	2.8	Whole
2a & b	GAE	0.96	0.44	7.8	2 whole filters	GAE	6.66	4.82	2.7	½ each filter
3a & b	GSA	-0.12	0.04	5.8	2 whole filters	GSA	4.06	4.99	2.7	½ each filter
4a & b	Quartz	-1.40	-	-	-	Quartz	-5.16	-	-	-
5a & b	GF/A	-	-	-	-	GF/A	-	-	-	-
5a & b	GF/D	0.00	0.05	6.6	2 whole filters	GF/D	-0.04	0.13	5.9	2 whole filters
6a & b	RA 934 AH	0.04	0.02	5.9	2 whole filters	RA 934 AH	0.08	0.14	5.6	2 whole filters

TABLE XIX
(CONTINUED)

Run Number		BRSP-13 Mass Gain (mg)	Sulfate (mg)	pH	Filter Portion Analyzed
1b	GA	0.12	0.03	6.0	Whole
2a & b	GAE	1.08	0.32	5.8	2 whole filters
3a & b	GSA	0.10	0.05	5.7	2 whole filters
4a & b	Quartz	0.66	Negligible	3.3	2 whole filters
5a & b	GF/A	-	-	-	-
5a & b	GF/D	0.18	0.06	6.4	2 whole filters
6a & b	RA 934AH	0.32	0.05	5.5	2 whole filters

For a given temperature, the filters seem to "saturate" and not gain much additional mass after a period of time (2-6 hours).

Use of Laboratory Preconditioned Filters in the Field

Two techniques were employed for laboratory preconditioning of filter media. One was an exposure to a controlled mixture of air, water and SO_2 at elevated temperature, as described in the preliminary laboratory screening tests. The other method was a sulfuric acid wash as previously described which is detailed in Appendix A. In the following paragraphs, those filters treated by the former method are termed "laboratory conditioned" and the filters for which the second method was used are referred to as "acid washed" filters.

In the field tests, unconditioned, laboratory conditioned, and acid washed glass fiber substrate materials were exposed to flue gases of differing temperatures. In order to obtain an estimate of what would be the maximum mass gains caused by flue gas, samples of various manufacturers glass fiber filter material were taken to the outlet of a hot side electrostatic precipitator (343°C, 650°F) and exposed to the flue gases for one week during a field test at steam plant C. Included in this sample were 27 Reeve Angel 934AH, 47 mm filters which were laboratory conditioned at SoRI for 12 hours. An alundum thimble holder was used to hold the samples and was placed in the flue gas stream on a probe preceded by a Gelman 47 mm stainless steel filter holder. The flow rate used was 0.4 ACFM and the pressure drop was approximately 10 inches of mercury, indicating that the flue gas was passing through the filter stack (81-47 mm filters). The inside diameter of the thimble holder is almost exactly 47 mm, insuring a good fit with the filters.

Table XX shows the mass changes recorded in this experiment. The filters are identified by manufacturer, name, and batch. Three observations can be made immediately:

1. Reeve Angel filter material conditioned at SoRI behaves as unconditioned Reeve Angel material behaves.
2. Reeve Angel filter material shows the lowest mass gains of all the filter materials exposed to the flue gas.
3. The Gelman Quartz material lost weight.

Since the Reeve Angel 934AH material, which was laboratory preconditioned, gained mass this means that such a procedure might not be useful. On the other hand, the laboratory conditioning was done at 232°C (450°F) and the flue temperature was 343°C (650°F). We know from previous work[1] that there is a strong dependence on temperature when mass gains occur. Other factors are that the exposure time was one week and that the filters were not handled during the test.

TABLE XX

Mass Changes Recorded in 47 mm Glass Fiber Filters
Exposed to 343^{o}C (650^{o}F) Flue Gas for One Week

Filter Manufacturer	Name	Batch No.	Number in Sample	Average Mass Change/ Filter (mg)	Percent Mass Change
Reeve Angel*	934AH	3307	27	0.89	0.79
Reeve Angel	934AH	3307	27	0.90	0.81
Reeve Angel	934AH	3307	3	0.91	0.82
Reeve Angel	934AH	4292	20	0.58	0.55
Whatman	GF/A	3563	3	6.19	7.0
MSA	1106BH	J888	3	12.06	10.9
Gelman	Spectroglass fiber	8192-20232	3	16.36	12.3
Gelman	AE	8204	5	17.36	13.1
Gelman	AE	8206	5	18.10	13.3
Gelman	Quartz	8198	5	-2.53	-1.6

---

* Conditioned at SRI for 12 hours.

The mass gains reported in Table XX probably are good maximum values. Previous experience with Reeve Angel 934AH material shows no mass gains as large as those recorded in the week's exposure. This last point will be discussed in greater detail later in this report.

Table XXI shows the results of chemical analyses made on 47 mm glass fiber substrate materials exposed to hot side electrostatic precipitator (ESP) flue gases and laboratory simulated flue gases. These substrates were prepared in various ways. Some were conditioned at SoRI, and some were conditioned at the hotside ESP. Others were not exposed to flue gases but analyzed in an unused state. Comparison with Table XX shows that the mass gains recorded were almost exclusively due to sulfate compounds formed on the substrates.

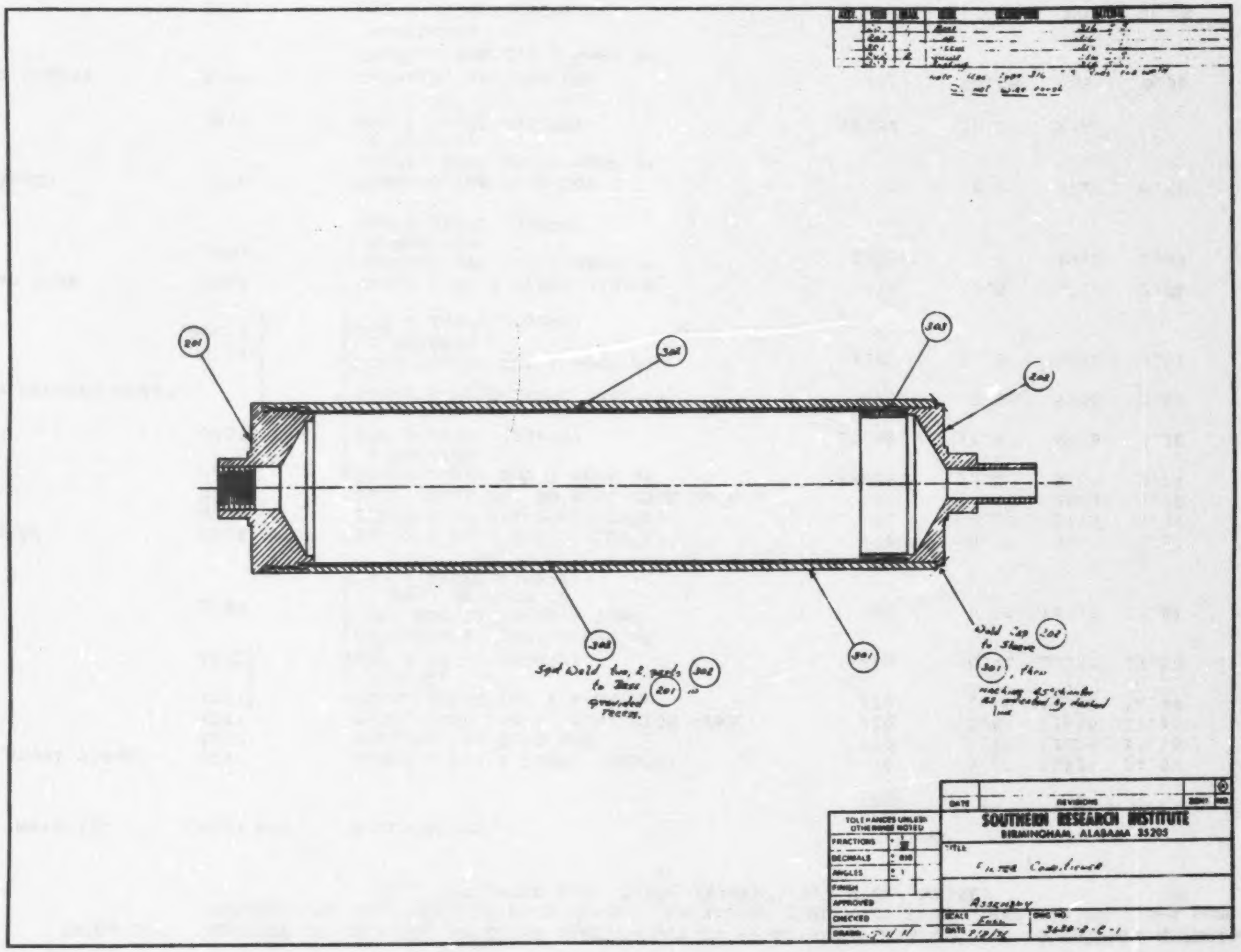
Of interest is the fact that calcium and magnesium content appear to be unrelated to sulfate mass gains. Filters which were washed in sulfuric acid solutions and then in distilled water and ethanol(ETOH) showed no significant amount of sulfate content. This is perhaps due to the washing out of sulfates, which were formed by the sulfuric acid, by the water and ETOH. In subsequent conditioning of the acid washed filter media, mass gains occurred which were much less than that caused in unconditioned substrates.

Since the sulfate mass gain by glass fiber substrates when they are exposed to hot flue gases is well documented, one approach to passivation is to expose or condition substrates to these gases prior to use in an impactor. Figures 20 through 22 show a conditioning chamber designed at SoRI. It is fabricated from 316 stainless steel which has been treated in hot nitric acid to remove any iron near metal surfaces. This chamber will hold enough Andersen substrates for a typical field test of one week duration. The chamber, with a Gelman 47 mm prefilter, is inserted into an outlet port and the enclosed substrates are exposed to filtered flue gas at 0.5 ACFM for periods from several hours to several days. Table XXII shows the averaged stage mass gains from blank Andersen and Brink impactor runs made at a variety of flue gas sources with Reeve Angel 934AH substrate material. These substrates were conditioned in hot flue gas for varying lengths of time. In no case were the stage mass gains as great as those found in the week-long exposure test (Table XX). Note than an Andersen substrate is typically one and one-half times as massive as a 47 mm filter (∿150 mg as opposed to 100 mg for Reeve Angel 934AH).

If preconditioning is to be useful then it is desirable to reduce the blank mass gains below these reported in Table XXII, as close to zero as possible. Figure 12 shows that a sulfuric acid wash reduces laboratory induced mass gains in Gelman AE material by a factor of 4. A sulfuric acid treatment of Reeve Angel 934AH material similarly should reduce mass gains in this substance.

TABLE XXI RESULTS OF CHEMICAL ANALYSES CARRIED OUT ON 47 mm GLASS FIBER FILTER SUBSTRATE MATERIALS EXPOSED AND UNEXPOSED TO FLUE GASES, LABORATORY SIMULATED FLUE GASES (1% SO_2, 3-5 ppm SO_3, SATURATED H_2O, 220°C (428°F)) and H_2SO_4 WASHES

Filter Material	Batch No.	Preparation	SO_4 (μg)	SO_4 %	Ca (μg)	Ca %	Mg (μg)	Mg %
Reeve Angel 934AH	3307	Baked 4 hr. @ 260°C (500°F)	<70	0.08	15237	24.52	2300	4.49
	4292	Unbaked, as from box	<70	0.10	13625	17.96	2362	3.78
	4292	H_2SO_4 wash for 90 min, ETOH wash	<70	<0.05	12900	17.10	2516	4.05
	3307	Conditioned for 1 week at Hotside	720	0.60	13380	16.49	2235	3.34
	4292	ESP @ 343°C (650°F)	468	0.40	13287	17.59	2537	4.08
	3307	Laboratory conditioned at SRI for 12 hours. Then 1 week at Hotside ESP @ 343°C (650°F)	720	0.60	14325	17.81	2362	3.56
Gelman AE	8204	Baked 2 hr @ 260°C (500°F)	<70	<0.05	5887	6.33	2212	2.88
	8206	Baked 4 hr @ 260°C (500°F)	<70	<0.05	5775	6.36	2212	2.96
	8206	H_2SO_4 Wash for 90 min, ETOH wash	<70	<0.05	5600	5.80	2322	2.93
	8206	Conditioned for 1 week at	18648	12.2	5705	5.24	2200	2.45
	8204	a Hotside ESP @ 343°C (650°F)	17640	11.9	5705	5.38	2200	2.52
Gelman Spectro Glass	8192-20232	Baked 2 hr @ 260°C (500°F)	<70	<0.05	5200	5.52	2226	2.87
		Conditioned for 1 week at a Hotside ESP @ 343°C (650°F)	17208	11.5	3690	3.51	2200	2.54
Whatman GF/A	3563	Baked 2 hr @ 260°C (500°F)	<70	<0.05	1320	2.08	56	0.11
	3563	Conditioned for 1 week at a Hotside ESP @ 343°C (650°F)	6552	6.9	1260	1.87	85	0.15
MSA 1106BH	J888	Unbaked, as from box	<70	<0.05	3725	4.60	1355	2.03
	J888	Conditioned for 1 week at a Hotside ESP @ 343°C (650°F)	12492	10.2	N/A		N/A	
Gelman Quartz	8198	Unbaked, as from box	<70	<0.05	425	0.38	137	0.15
	8198	Conditioned for 1 week at a Hotside ESP @ 343°C (650°F)	1044	0.7	420	0.38	132	0.14


201
302
303
202
Weld cap 202 to Sleeve 301, then machine 45° chamfer as indicated by dashed line
301
303
Spot Weld two, 2 parts 302 to Base 201 in provided recess.
TOLERANCES UNLESS OTHERWISE NOTED
FRACTIONS
DECIMALS
ANGLES
FINISH
APPROVED
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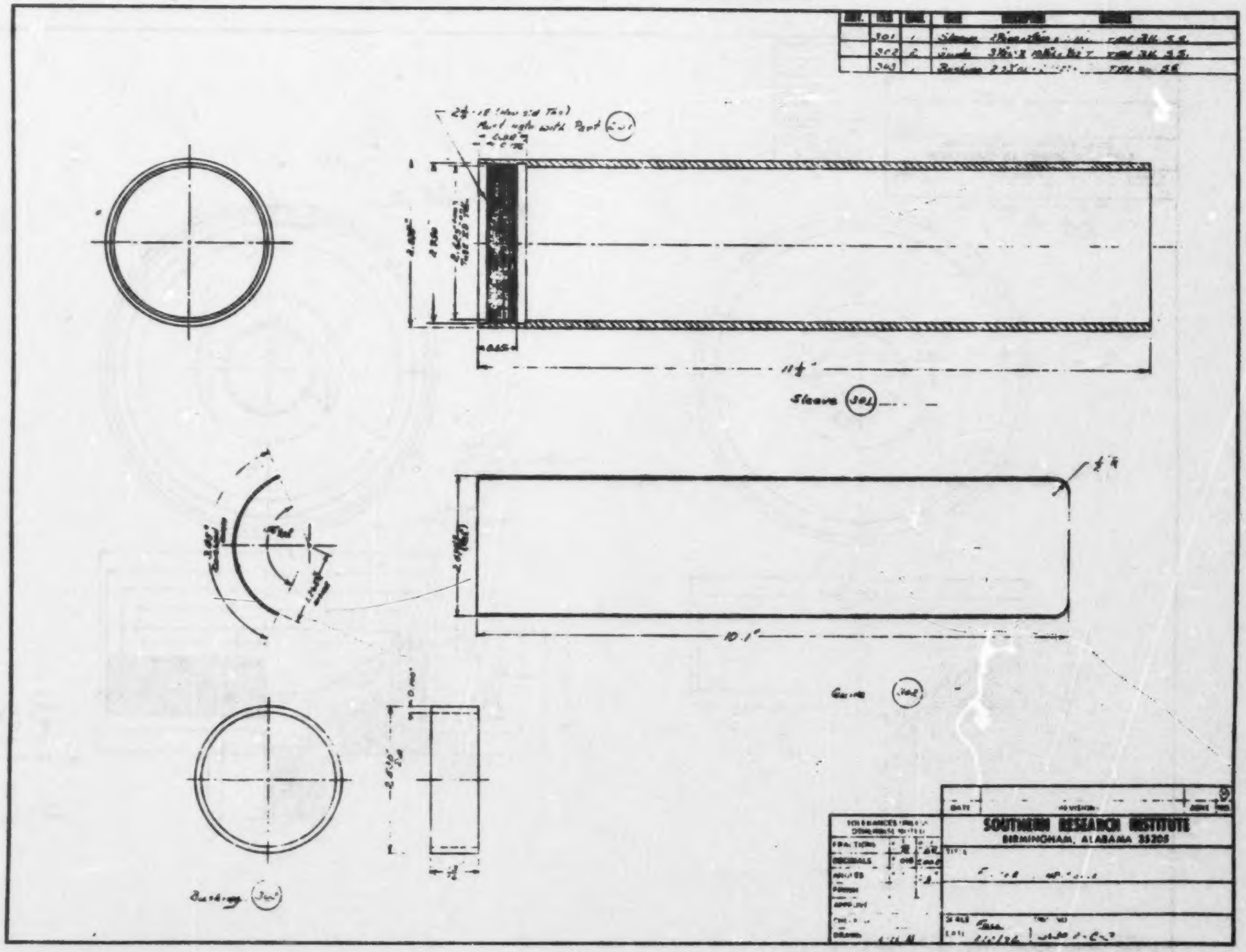
DATE
REVISIONS
SOUTHERN RESEARCH INSTITUTE
BIRMINGHAM, ALABAMA 35205
TITLE
Filter Conditioner
Assembly
SCALE
DATE
DWG NO.
3630-8-C-1

Figure 20. Substrate conditioning chamber, side view.


301
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303
Sleeve
Guide
Bushing
11¼"
10.1"
SOUTHERN RESEARCH INSTITUTE
BIRMINGHAM, ALABAMA 35205

Figure 21. Substrate conditioning chamber, interior components.

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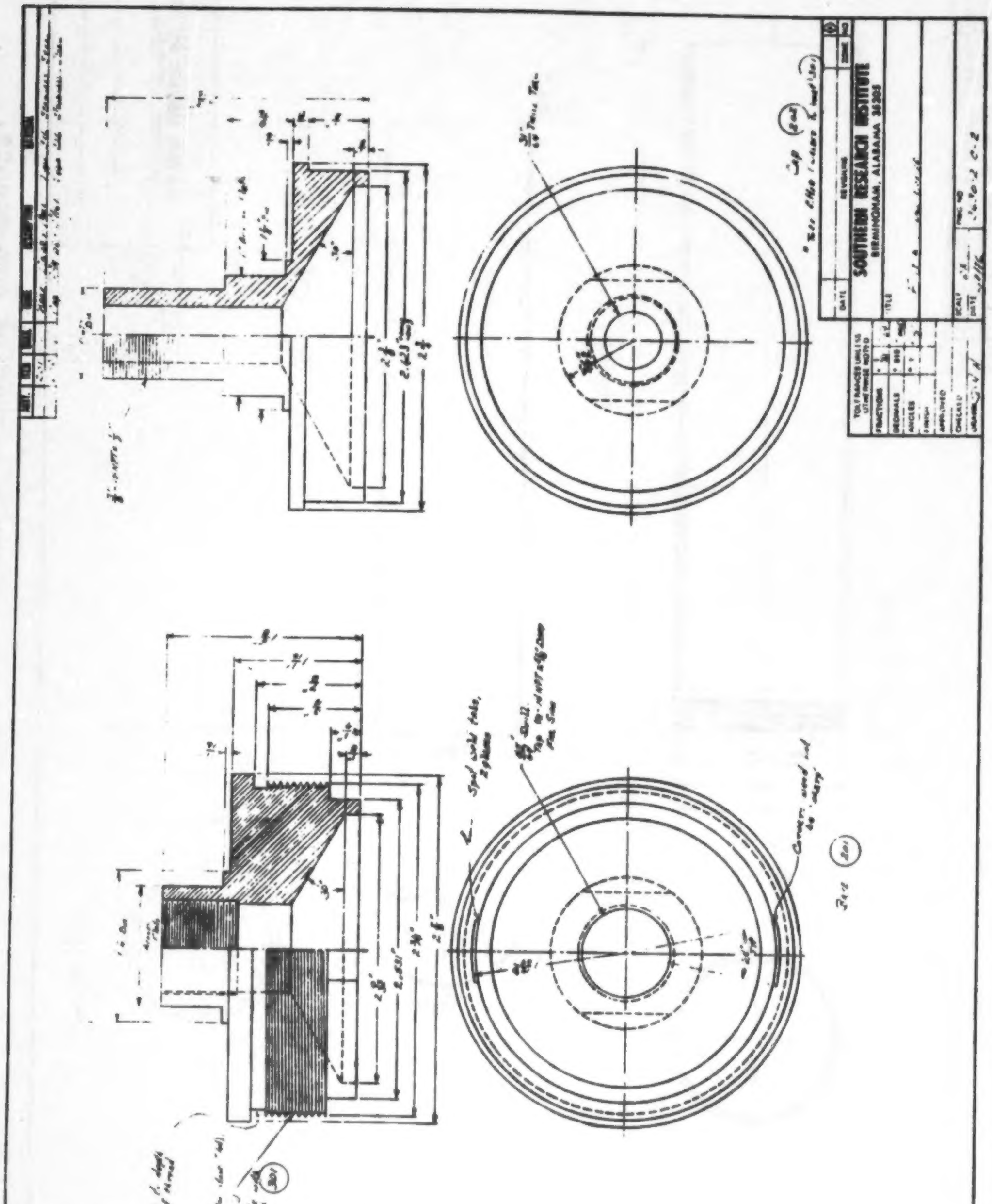
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Figure 22. Substrate conditioning chamber, end caps.

TABLE XXII

Averaged Stage Mass Gains for "Blank" Impactor Runs with Reeve Angel 934AH Glass Fiber Filter Substrate Material[1]

Source	Temperature (°C)	Temperature (°F)	Preconditioning Time (hours)	SO_2 Concentration (ppm)	Run Time (hours)	Average Mass Gain per Stage (mg)
Aluminum Smelter (Scrubber)	49	120	0	<1	3.8	-0.03
					1.75	-0.05
Power Plant (Utah)	149	300	24	330	16	0.30
Power Plant (St. Louis)	166	330	8	~2000	0.5	0.24
Paper Mill (Kraft Recovery)	177	350	12	95	.80	0.14
	177	350	12	107	1.60	0.13
Power Plant (Pennsylvania)	138	280	15	760	0.50	0.20
	138	280	5	760	0.50	0.43
Cement Kiln	104	220	0	<1	2.0	0.15
Power Plant (Alabama)	160	320	12	3500	2.0	0.49
	160	320	12	3500	4.0	0.06
	160	320	12	3500	1.5	0.28
Power Plant (North Carolina)	327	620	12	900	6.0	0.41
	327	620	12	900	2.0	0.18
	338	640	12	900	2.0	0.25
	338	640	0	900	2.0	0.63
	338	640	12	900	0.4	0.02
	338	640	12	900	0.2	0.08[2]
Power Plant (Tennessee)	154	310	12	2500	0.65	0.03[2]
	160	320	12	2500	4.0	0.16

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1. Stage mass gains are averaged for each condition. Usually there is more than one blank run for any given temperature.

2. Brink impactor blank run, all others are Andersen impactor blank runs.

Experiments were conducted in which unconditioned, laboratory conditioned, and acid treated glass fiber filter material were to be exposed to flue gases at an electric power generating station, steam plant D. This plant has both hot-side and cold-side electrostatic precipitators operating off boilers fired with the same coal. Flue gases at the hot-side outlet and cold-side outlet are hotter and colder, respectively, than the laboratory conditioning temperature. Two trips were made to this plant for the purpose of exposing glass fiber filter material to filtered hot- and cold-side flue gases in simultaneous runs.

Table XXIII shows the mass gains which occurred during the first trip when different kinds of 47 mm glass fiber filter substrate media were exposed to flue gases in the hot- and cold-side precipitators. The filters were subdivided into three broad classes: (1) unconditioned filters, (2) filters conditioned in the laboratory at SoRI, (3) acid washed filters. Two identical stacks of filters were made up and placed in identical stainless steel alundum filter holders. Twelve different samples were placed in each filter holder in the order and numbers given in Table XXIII. For example, 20 Reeve Angel 934AH filters which had been laboratory conditioned at SoRI for a total of 26 hours were at the top in each filter holder, and 10 Gelman AE sulfuric acid washed filters were at the bottom of each filter holder.

These filter holders, loaded with filters, were exposed for six hours each at the hot- and cold-side precipitators. Flow rates were adjusted to pull total volume of gas (reduced to standard conditions) through each group of filters. The cold-side temperature was 143°C (290°F) and the hot-side temperature was 321°C (610°F). Filters conditioned at SoRI were conditioned at 221°C (430°F).

Several comments may be made about the results presented in this table:

1. The Reeve Angel 934AH filter material which was washed in sulfuric acid, rinsed in distilled water and ethanol (ETOH), and then laboratory conditioned for 18 hours at SoRI had the lowest mass gains of all materials tested: zero for the cold side, and -0.01 mg/filter for the hot side.
2. Material which had been conditioned at SoRI (26 or 18 hours) and then exposed to flue gases in the hot-side precipitator generally lost weight. This may be due to handling losses as some black particulate matter was inadvertantly allowed to fall in among the filters when the Gelman prefilter was removed from the front of the filter holder, and some of these filters had to be dusted off. This was done with a soft camels' hair brush.

TABLE XXIII MASS GAINS OF 47 mm GLASS FIBER FILTER SUBSTRATE MATERIALS EXPOSED TO FLUE GASES IN HOT AND COLD SIDE PRECIPITATORS[1] AT GORGAS STEAM PLANT, 25 June, 1976

Position in Thimble Holder	Filter Type	Batch No.	No. Filters	Type of Pre-conditioning	Mass Before Exposure (grams)		Mass After Exposure (grams)		Mass Increase/Filter (mg)		Percent Mass Gain	
					Hot Side	Cold Side	Hot Side	Cold Side	Hot Side	Cold Side	Hot Side	Cold Side
1	Reeve Angel 934AH	3307	20	26 Hours, Lab	2.172	2.1894	2.2189	2.1919	0.085	0.125	0.08	0.11
2	Reeve Angel 934AH	4292	20	None	2.1003	2.1019	2.1047	2.1037	0.190	0.090	0.18	0.09
3	Gelman AE	8204	20	26 Hours, Lab	2.8388	2.8748	2.8331	2.8823	-0.285	0.375	-0.20	0.26
4	Gelman AE	8204	20	None	2.5781	2.7365	2.7013	2.7905	6.160	2.700	4.78	1.97
5	Gelman Spectro Glass	8292-20232	20	26 Hours, Lab	2.8171	2.8030	2.8030	2.8228	-0.165	0.285	-0.12	0.20
6	Gelman Spectro Glass	8292-20232	20	None	2.6852	2.7493	2.743	2.6961	2.955	0.545	2.20	0.41
7	Whatman GF/A	3563	20	26 Hours, Lab	1.8278	1.8355	1.8200	1.8389	-0.390	0.170	-0.43	0.19
8	Whatman GF/A	3563	20	None	1.7095	1.7228	1.7374	1.7323	1.395	0.475	1.63	0.55
9	Reeve Angel 934AH	4292	10	H_2SO_4, H_2O-ETOH wash[3]	1.0476	1.0498	1.0475	1.0498	-0.010	0.000	-0.01	0.00
10	Gelman AE	8206	10	H_2SO_4, H_2O-ETOH wash[3]	1.3860	1.3851	1.3919	1.3887	0.590	0.360	0.43	0.19
11	Reeve Angel 934AH	4292	10	H_2SO_4, dry, H_2O wash	1.0515	1.0437	1.0478	1.0407	-0.370	-0.300	-0.35	-0.29
12	Gelman AE	8204	10	H_2SO_4, dry, H_2O wash	1.2357	1.2415	1.2528	1.2475	1.710	0.600	1.38	0.48

[1] Hot Side = 321°C (610°F), Cold Side = 143°C (290°F)
[2] After 24 hours desiccation
[3] Preconditioned at SRI for 18 hours

3. In general, filter material which has been conditioned at SoRI, acid washed, or both, showed much less mass change per filter than unconditioned filter media.
4. Unconditioned filters gained more mass when exposed to hot-side precipitator flue gases than when exposed to cold-side precipitator flue gases. This was expected from previous experience..

Table XXIV shows the mass gains which occurred during the second trip. The acid treated Reeve Angel 934AH and Whatman GF/A material exposed during this trip were prepared according to the following procedure: One hundred-fifty 47 mm filters were conditioned in a 50-50 mixture by volume of distilled water and sulfuric acid at 88°C (190°F) for one hour. The filter stack was then separated into three groups of 50. All three groups were washed first in distilled water and then ethyl alcohol (ETOH). Finally, one group was rinsed in distilled water; one group was rinsed in isoprophyl alcohol (IPA), and the last group was rinsed in ETOH. After draining, each group was baked in a laboratory oven at approximately 104°C (220°F) for 6 hours.

Along with the laboratory conditioned material, unconditioned glass fiber and quartz fiber filter substrate materials were also exposed to the flue gases. These materials included Reeve Angel 934AH, Whatman GF/A, Gelman AE, Gelman SpectroGlass, Old Gelman Type A (pre-1975), and a quartz fiber material supplied by Mr. D. B. Harris, Project Officer of this contract.

The mass changes recorded in Table XXIV differ somewhat from those observed in the other test (first trip). All acid washed substrates lost mass. But, examination of unused, acid treated substrates prepared for this test showed that the mass losses were due to inadequate washing of the filter media after acid treatment. Samples of the Whatman GF/A material which showed a hot-side loss of 2.29 mg/filter were placed in a nearly neutral distilled water solution (pH = 6.0). After mixing for several minutes the pH of the filter-water slurry had dropped to 4.4. Other samples of these filters which had been exposed to air for several weeks showed 2 to 3 mg/filter mass losses upon baking at 100°C (212°F) for 2 hours. When exposed to ambient air after baking, these filters regained 1 to 2 mg/filter of the baking weight loss. The high boiling point of sulfuric acid (338°C, 640°F) means that the usual bakeout (100°C-200°C) of substrates following acid treatment will be insufficient to remove most residual acid. To be sure that most, if not all, of the acid is removed after treatment, the pH of the substrate water should be compared with that of the distilled water used for washing. Wash water should be left in the container holding the substrates for several minutes prior to making a pH determination.

The unconditioned material behaved much like that exposed on the first trip. The Gelman Type A (pre-1975) material gained mass

TABLE XXIV MASS GAINS OF 47 mm GLASS AND QUARTZ FIBER FILTER SUBSTRATE MATERIALS EXPOSED TO FLUE GASES IN HOT AND COLD SIDE PRECIPITATORS[1] AT STEAM PLANT D, 31 AUGUST - 1 SEPTEMBER 1976

Position in Thimble Holder	Filter Type	Batch No.	No. Filters	Type of pre-conditioning	Mass Before Exposure (grams) Hot Side	Mass Before Exposure (grams) Cold Side	Mass After Exposure[2] (grams) Hot Side	Mass After Exposure[2] (grams) Cold Side	Mass Change/Filter (mg) Hot Side	Mass Change/Filter (mg) Cold Side	Percent Mass Change Hot Side	Percent Mass Change Cold Side
1	Reeve Angel 934AH	4292	20	None	2.1006	2.0945	2.1033	2.0942	0.135	-0.015	0.13	-0.01
2	Whatman GF/A	3563	20	None	1.7051	1.7089	1.7277	1.7141	1.130	0.260	1.33	0.30
3	Quartz[3]	-	4	None	0.6212	0.6238	0.6079	0.6146	-3.325	-2.300	-2.14	-1.47
4	Gelman Type A[4]	-	4	None	0.5484	0.5527	0.5560	0.5546	1.90	0.475	1.39	0.34
5	Gelman Type AE	8204	8	None	1.0638	1.0653	1.1050	1.0752	5.15	1.24	3.87	0.93
6	Gelman Spectro Glass	8292-20232	8	None	0.9846	0.9990	1.0184	1.0033	4.225	0.538	3.43	0.43
7	Reeve Angel 934AH	4292	20	$H_2SO_4-H_2O$	2.1278	2.1061	2.0954	2.0959	-1.620	-0.510	-1.52	-0.48
8	Reeve Angel 934AH	4292	20	H_2SO_4-IPA	2.0939	2.0931	2.0920	2.0882	-0.095	-0.245	-0.09	-0.23
9	Reeve Angel 934AH	4292	20	H_2SO_4-ETOH	2.1020	2.0841	2.0961	2.0812	-0.295	-0.145	-0.28	-0.14
10	Whatman GF/A	3563	20	$H_2SO_4-H_2O$	1.6797	1.6761	1.6676	1.6689	-0.605	-0.360	-0.72	-0.43
11	Whatman GF/A	3563	20	H_2SO_4-IPA	1.7374	1.7675	1.7250	1.7350	-0.620	-1.625	-0.71	-1.84
12	Whatman GF/A	3563	20	H_2SO_4-ETOH	1.7389	1.7125	1.6931	1.6823	-2.29	-1.510	-2.63	-1.76

Notes:

[1] Hot Side = 321°C (610°F), Cold Side = 143°C (290°F)
Exposure time was 6 hours at 0.35 ACFM.

[2] After 24 hours desiccation.

[3] Supplied by Project Officer.

[4] Old Gelman Type A glass fiber filter material (pre 1975).

but not as much as contemporary Gelman AE. Finally, the quartz fiber material lost a considerable amount of mass. This material is much stronger than the Pallflex Tissuquartz tested earlier but is still fragile. One final hot-side, cold-side exposure test was planned at steam plant D, but boiler outages forced the test to be moved to another source. During November, 1976, a field test was executed at steam plant E, and some substrates were prepared for flue gas exposure tests there.

Differently preconditioned 47 mm glass fiber filter materials were exposed for 8.5 hours to 321°C (609°F) flue gas. Gas flow through the filter stack was 0.3 ACFM. An attempt was made to expose a similar set of preconditioned 47 mm filters to flue gas after the air preheater (117°C, 242°F), but this attempt failed when the filters were moistened.

Table XXV shows the recorded mass changes. Reeve Angel 934AH shows almost negligible mass changes on a "per filter" basis, regardless of conditioning. On the other hand, the Whatman GF/A material shows small mass gains for sulfuric acid wash preconditioning and much larger gains with no pre-treatment. The acid washed filters were conditioned in a 50-50 (by volume) solution of concentrated sulfuric acid and distilled water at a temperature of 115°C (239°F) for two hours. After this treatment, the filters were rinsed with distilled water until the pH of the rinse water from the filters (5.0) was nearly the same as the pH of the distilled water (5.5). Some of the filters were further rinsed in isopropanol or ethanol. The filters were next spread out and allowed to dry in an oven at 104°C (220°F). When dry they were baked at 228°C (550°F) for four hours to drive off any remaining sulfuric acid. Then the filters were desiccated until use. Adequate rinsing in distilled water is crucial to the conditioning of these filters. Otherwise residual sulfuric acid will be driven off by the flue gas if above 650°F and the filters will show a mass loss.

The low mass changes for Reeve Angel 934AH material reported in Table XXV are at variance with some of the mass changes recorded for blank Andersen and Brink impactor runs shown in Table XXVI. Table XXVI shows average mass gains per filter for Andersen and Brink impactor blank runs at two flue gas sources. These sources were the steam plant E, which has a hot-side precipitator operating from 316° to 372°C (600° to 700°F), and the steam plant F with a cold-side electrostatic precipitator operating from 93° to 121°C (200° to 250°F). The acid washed material used here was prepared as indicated above with careful attention to adequate rinsing.

The average mass of an Andersen impactor glass fiber substrate is near 150 mg. Scaling up the mass changes seen in Table XXV for 47 mm Reeve Angel 934AH acid washed material gives an average of 0.05 to 0.07 mg per Andersen substrate. Most of the blank mass gains of Andersen substrates reported in Table XXVI are much larger

TABLE XXV. MASS GAINS OF 47 mm GLASS FIBER FILTER MATERIALS EXPOSED TO FLUE GASES IN A HOT SIDE PRECIPITATOR[1] AT STEAM PLANT E, NOVEMBER 1976.

Position in Thimble Holder	Filter Type	Batch No.	No. Filters	Type of Preconditioning	Initial Weight (grams)	Final Weight (grams)	Mass Change/ Filter (mg)	Percent Mass Change
1	Reeve Angel 934AH	4292	20	none	2.0923	2.0915	-0.040	-0.04
2	Whatman GF/A	3563	20	none	1.7432	1.7640	1.040	1.19
3	Reeve Angel 934AH	4292	20	Flue gas at Gorgas[2], 6 hrs	2.1025	2.1010	-0.075	-0.07
4	Reeve Angel 934AH	4292	20	H_2SO_4-H_2O-Isopropanol	2.0996	2.1005	0.045	0.04
5	Whatman GF/A	3563	20	H_2SO_4-H_2O-Isopropanol	1.6823	1.6852	0.145	0.17
6	Reeve Angel 934AH	4292	20	H_2SO_4-H_2O-Ethanol	2.0757	2.0764	0.035	0.03
7	Whatman GF/A	3563	20	H_2SO_4-H_2O-Ethanol	1.7971	1.8001	0.150	0.17
8	Reeve Angel 934AH	4292	20	H_2SO_4-H_2O	2.0728	2.0735	0.035	0.03
9	Whatman GF/A	3563	20	H_2SO_4-H_2O	1.8218	1.8258	0.200	0.22

NOTES:
1. Exposure time was 8.5 hours @ 321°C (609°F) Gas flowrate was 0.3 ACFM
2. Six hours at 321°C (610°F). Gas flowrate was 0.35 ACFM

TABLE XXVI Blank Impactor Run Mass Gains at Two Different Flue Gas Sources, Reeve Angel 934AH Glass Fiber Substrate Material

Type of Impactor	Location	Type of Preconditioning[1]	Temperature (°C)	Temperature (°F)	Flowrate (ACMM)	Flowrate (ACFM)	Run Time (min)	Number of Blank Runs of this type	Average Mass Gain (mg)	Standard Deviation of Mass Gain (mg)
Andersen	Plant F[2] (pretest)	Untreated	121	250	0.012	0.43	360	1	-0.179	0.124
Andersen	Plant F (pretest)	In Situ Conditioned	110	230	0.015	0.53	360	3	0.293[3]	0.234[3]
Andersen	Plant F (pretest)	H_2SO_4	102	215	0.010	0.35	240-360	2	0.061	0.111
Andersen	Plant F (pretest)	H_2SO_4 wash, In Situ conditioned	102	215	0.014	0.5	360	2	0.124	0.035
Andersen	Plant F (test)	H_2SO_4 wash, In Situ conditioned	102	215	0.014	0.5	150-252	5	0.169	0.099
Brink	Plant F (test)	H_2SO_4 wash, In Situ conditioned	101	214	0.0008	0.03	45	4	-0.020	0.077
Andersen	Plant E[4] (Test 1)	Untreated	343	650	0.011	0.4	240	2	0.394	0.143
Andersen	Plant E (Test 1)	H_2SO_4 wash	343	650	0.011	0.4	240	2	0.659	0.255
Andersen	Plant E (Test 1)	H_2SO_4 wash, In Situ conditioned	343	650	0.011	0.4	240	3	0.572	0.106
Andersen	Plant E (Test 2)	H_2SO_4 wash, 370°C Bake In Situ conditioned	316	600	0.017	0.6	82-210	7	-0.019	0.091
Brink	Plant E (Test 1)	H_2SO_4 wash, In Situ conditioned	357	675	0.0008	0.03	30	4	0.008	0.061
Brink	Plant E (Test 2)	H_2SO_4 wash, 370°C Bake, In Situ, conditioned	357	675	0.001	0.04	30-45	8	0.098[3,5]	0.080[3,5]

Notes

1. In Situ conditioning is usually for 24 hours
2. Steam Plant F
3. After elimination of three outlier points
4. Steam plant E
5. Blank mass gains may be unusually large because of rubber o-ring seals melting and outgassing

than this with the important exception of the data from steam plant E, Test 2. Substrates for this test were prepared as described above but were baked at 370°C (700°F) for one-half hour. Mass gains by these substrates are uniformly low and quite acceptable. Blank substrates in Brink impactors showed larger mass gains as compared to Test 1, but during Test 2 silicone rubber O-rings were used for sealing, and these O-rings disintegrated on disassembly of the impactor causing fouling of the substrates. The good set of data from Test 2, steam plant E, for the Andersen blank impactor runs does not necessarily indicate that the problem of substrate mass gains due to flue gas reactions has been solved. What it does show is that acid washing, baking, and *in situ* conditioning can provide a lowered average mass gain coupled with a much lower standard deviation of mass gain. The lower standard deviation of mass gain is important in itself since it means that the substrates all behave in a similar manner.

Summary of Results of Evaluation of Filter Media

Untreated filter materials used as impactor substrates will almost invariably increase in mass when subjected to the hot flue gases normally encountered in field applications. Conversion of SO_2 to various sulfates appears to be the cause of mass gains. The various filter materials tested vary widely in the amount of mass change which occurs under a particular set of flue gas conditions.

Preconditioning techniques can be used to force the production of sulfates in a filter medium, leaving a minimal number of sites available for chemical reaction in the flue gas, and hence, providing substrate material for which minimum mass gains occur during use in an impactor. The best results were achieved when substrates were washed in sulfuric acid, following the procedure detailed in Appendix A, baked, and conditioned *in situ*.

Of the filter materials studied only the Reeve Angel 934AH was found to be suitable in all respects for use as cascade impactor substrates.

SECTION 3

CONCLUSIONS AND RECOMMENDATIONS

Collection stages of most types of cascade impactors are very heavy in comparison with the amounts of particulate material normally collected. It is therefore the usual practice to augment each collection stage with a lightweight substrate to improve weighing accuracy. Generally, two classes of substrates are used -- greased metal foils, and fibrous filter material.

Greased foils provide resistance to particle bounce and scouring effects, but greases tend to be unstable at elevated temperatures. Some tend to harden, and in others the viscosity may become reduced so that they may flow or be blown off the surface by the high velocity gas flowing through the impactor. Of the greases tested, Apiezon H was found to perform most satisfactorily. This grease may be used at temperatures up to approximately 177°C (350°F). No greases were found to be useable at higher temperatures.

Mass gains exhibited by glass fiber filter materials when they are exposed to the SO_x components in flue gas streams pose a complicated problem. Experiments show that these mass gains are caused by formation of sulfates due to a gas phase reaction with SO_x. Laboratory and field experiments indicate that the only glass fiber filter material suitable for use as a cascade impactor substrate is Reeve Angel 934AH. When this material is acid treated, according to a procedure given in Appendix A, mass gains caused by flue gas reactions can be kept quite small.

It is recommended that acid washing, baking and in situ conditioning be used whenever large blank mass gains with large standard deviations are expected. In this context, "large" refers to substrate mass gains greater than several tenths of a milligram.

Further research may provide a technique for passivating glass fiber materials to all mass gains. It has been suggested that a high temperature polymer or silicone compound might be developed to coat the glass fibers in much the same way that the Gelman SpectroGlass material is prepared for use at low temperatures.

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APPENDIX A

Procedure for Acid Washing of Substrates

1. Submerge the substrates to be conditioned in a 50-50 mixture (by volume) of distilled water and reagent grade concentrated sulfuric acid at 110°-115°C (230°-239°F) for 2 hours. This operation should be carried out in a hood with clean glassware. Any controllable laboratory hotplate is suitable.

The substrates may need to be weighted down to keep them from floating. For this purpose, place a teflon disc on the top and bottom of the substrate stack. The top disc can be held down with a suitable glass or teflon weight.

2. When the substrates are removed from the acid bath they should be allowed to cool to room temperature. They are next placed in a distilled water bath and rinsed continuously with a water flow of 10-20 cm^3/min. The substrates should be rinsed until the pH of the rinse water, on standing with the substrates, is nearly the same as that of the distilled water. The importance of thorough washing cannot be over-emphasized.

3. After rinsing in distilled water the substrates are rinsed in reagent grade isopropanol (isopropyl alcohol). They should be submerged and allowed to stand for several minutes. This step should be repeated four to five times, each time using fresh isopropanol.

4. Allow the substrates to drain and dry. They can be spread out in a clean dry place after they have partially dried (dry enough to handle).

5. When the filters are quite dry to the touch they should be baked in a laboratory oven to drive off any residual moisture or isopropanol. Bake the substrates at 50°C (122°F) for about two hours, at 200°C (392°F) for about two hours, and finally at 370°C (700°F) for about three hours. The substrates are now ready for in situ conditioning.

As a final check, place two substrates in about 50 ml of distilled water, and check the pH. The substrates to be checked for pH should be torn into small pieces, placed in the water, and stirred for about 10 minutes before the pH is measured. If the pH is significantly lower than that of the distilled water, then the filters should be baked out at 370°C (700°F) for several hours more to remove any residual sulfuric acid. The boiling point of sulfuric acid is 338°C (640°F), so high temperatures must be used.

Figure 23 is a flow chart representing the acid wash procedure described in the foregoing paragraphs.

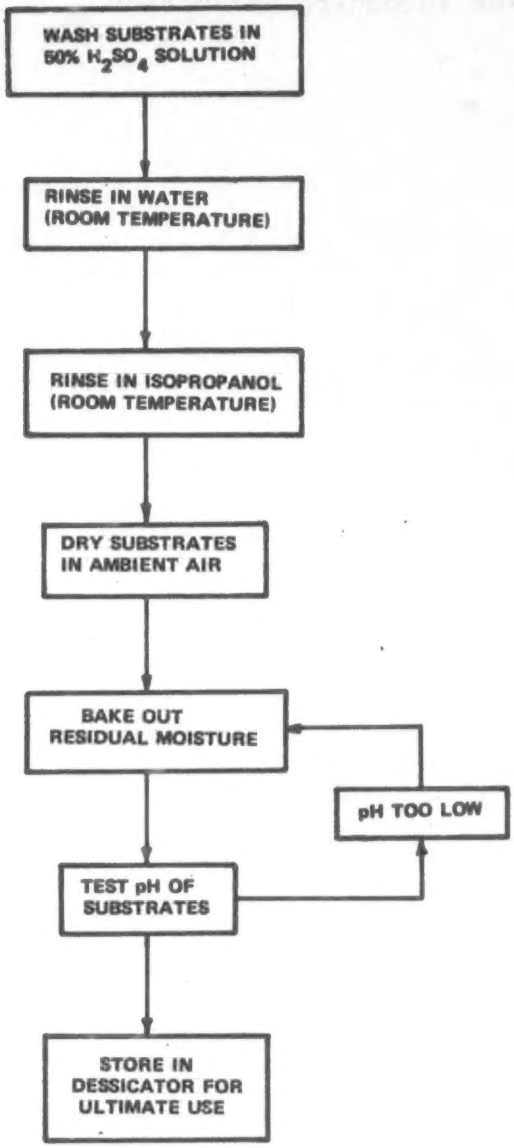
WASH SUBSTRATES IN 50% H_2SO_4 SOLUTION
RINSE IN WATER (ROOM TEMPERATURE)
RINSE IN ISOPROPANOL (ROOM TEMPERATURE)
DRY SUBSTRATES IN AMBIENT AIR
BAKE OUT RESIDUAL MOISTURE
pH TOO LOW
TEST pH OF SUBSTRATES
STORE IN DESSICATOR FOR ULTIMATE USE

Figure 23. Flow chart for acid wash treatment of glass fiber filter material.